

# CERTIFICATE OF ANALYSIS

## 14X HS3 (batch L)

### Certified Reference Material Information

Type: HIGH-SPEED STEEL (CHILL CAST)  
Form and Size: Disc 40mm Diameter x 17mm Thickness  
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>	0.855	0.318	0.046	0.035	0.621	0.706	5.25	1.24
Uncertainty <sup>2</sup>	0.011	0.016	0.002	0.002	0.010	0.017	0.05	0.03

Element	Cu	Co	W	V	As	Sn	Pb	N
Value <sup>1</sup>	0.200	10.64	17.93	1.79	0.044	0.288	0.010	0.087
Uncertainty <sup>2</sup>	0.003	0.07	0.15	0.02	0.003	0.008	0.001	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 \_\_\_\_\_

C Eveleigh

on 22<sup>nd</sup> November 2007



## **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. Metal was 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 the 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 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 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	0.826	0.284	0.0410	0.0310	0.597	0.661	5.140	1.180
2	0.835	0.287	0.043	0.0313	0.600	0.670	5.152	1.182
3	0.839	0.291	0.0433	0.0315	0.615	0.688	5.167	1.183
4	0.844	0.309	0.0452	0.0335	0.616	0.698	5.191	1.224
5	0.848	0.312	0.0459	0.0345	0.616	0.700	5.210	1.230
6	0.852	0.320	0.0460	0.0346	0.620	0.715	5.268	1.23
7	0.861	0.323	0.0470	0.0356	0.625	0.718	5.271	1.240
8	0.862	0.328	0.0472	0.0371	0.627	0.720	5.275	1.252
9	0.864	0.341	0.0472	0.0382	0.630	0.720	5.291	1.252
10	0.866	0.346	0.0478	0.039	0.640	0.737	5.327	1.255
11	0.882	0.352	0.051	0.039	0.650	0.739	5.359	1.284
12	0.885		0.0519	0.0392			5.370	1.285
13								1.298
<b>Mean</b>	<b>0.855</b>	<b>0.318</b>	<b>0.0464</b>	<b>0.0354</b>	<b>0.621</b>	<b>0.706</b>	<b>5.252</b>	<b>1.238</b>
<b>Std Dev</b>	0.018	0.024	0.0031	0.0031	0.016	0.025	0.079	0.039
<b>C<sub>(95%)</sub></b>	0.011	0.016	0.0020	0.0020	0.010	0.017	0.050	0.024

Sample	Cu	Co	W	V	As	Sn	Pb	N
1	0.194	10.50	17.68	1.73	0.0397	0.270	0.0092	0.0780
2	0.195	10.51	17.70	1.732	0.0400	0.278	0.0094	0.0858
3	0.195	10.52	17.73	1.752	0.0408	0.279	0.0097	0.0859
4	0.195	10.55	17.90	1.786	0.0410	0.280	0.0098	0.0870
5	0.199	10.58	17.92	1.791	0.0415	0.280	0.010	0.0880
6	0.199	10.61	18.01	1.794	0.0420	0.283	0.0111	0.0903
7	0.201	10.62	18.02	1.800	0.0441	0.283	0.0120	0.0926
8	0.201	10.62	18.07	1.801	0.0459	0.284	0.0121	
9	0.202	10.71	18.07	1.802	0.0491	0.295		
10	0.203	10.78	18.08	1.807	0.0513	0.299		
11	0.208	10.80	18.09	1.815	0.052	0.309		
12	0.208	10.82		1.82		0.311		
13				1.830				
<b>Mean</b>	<b>0.200</b>	<b>10.64</b>	<b>17.93</b>	<b>1.789</b>	<b>0.0443</b>	<b>0.288</b>	<b>0.0104</b>	<b>0.0868</b>
<b>Std Dev</b>	0.005	0.12	0.16	0.032	0.0046	0.013	0.0012	0.0046
<b>C<sub>(95%)</sub></b>	0.003	0.07	0.11	0.019	0.0031	0.008	0.0010	0.0042

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
IncoTest Ltd	Hereford, England	UKAS accreditation 0281
Bodycote Materials Testing	Middlesbrough, England	UKAS accreditation 0239
London & Scandinavian Met Co Ltd	Rotherham, England	UKAS accreditation 1091
Metals Technology (Testing) Ltd	Sheffield, England	UKAS accreditation 0963
Universal Scientific Laboratory Ltd	Milperra, NSW, Australia	NATA accreditation 0492
Institute of Iron & 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 Pvt Ltd	Mumbai, India	NABL accreditation 0367
Laboratory TUV-Nord Czech	Brno, Czech Republic	CAI accreditation 1060
De Bruyn Spectroscopic Solutions	Johannesburg, South Africa	
Genitest Inc	Montreal, Canada	

Note: to achieve National Accreditation (eg UKAS, NATA, CNAL, NABL, CAI), 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	1, 3, 4, 11	-	-	2, 6, 8-10	gravimetric (perchloric acid)
				5, 7	photometric (molybdenum blue)
Sulfur	-	-	-	all	combustion (infra-red detection)
Phosphorus	2, 4-7, 10	1	-	3, 8, 11, 12	photometric (molybdenum blue)
				9	volumetric (alkalimetric)
Manganese	3-5, 7, 8, 10	11	6	1, 9	photometric (periodate)
				2	volumetric (arsenite, ammonium nitrate)
Nickel	5, 6, 9-11	8	1, 2, 3, 4	7	photometric (dimethyl glyoxime)
Chromium	3, 5, 9-11	12	7	1, 2, 4, 6, 8	volumetric (ferrous ammonium sulfate)
Molybdenum	3, 7, 8, 11-13	6	2, 5	1, 4, 9, 10	photometric (thiocyanate)
Copper	2, 3, 8, 9, 11	-	1, 4-7, 12	10	photometric (BCO)
Cobalt	1, 6, 9-12	7	2, 3, 8	4	gravimetric
				5	photometric (2-β-naphthol)
Tungsten	1, 6-9	11	-	2, 10	photometric (thiocyanate)
				4, 5	gravimetric
				3	photometric (tin dichloride)
Vanadium	3, 5-7, 10-13	-	1, 2, 8, 9	4	volumetric (ferrous ammonium sulfate)
Arsenic	1, 4-7, 9-11	-	2, 3	8	photometric (molybdenum blue)
Tin	2-4, 6, 9-11	5	1, 7, 8, 12		
Lead	1, 4-7	-	2, 3, 8		
Nitrogen	-	-	-	1-4, 6, 7	inert gas fusion (thermal conductivity)
				5	photometric (Nessler reagent)

## 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).

The unidirectional solidification effects associated with chill casting have led to minor segregation to the rear of the disc. The above certification is therefore only applicable from the front face of the disc, to a depth of 12mm. The remainder, ~5mm, is not certified

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 November 2027, 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.