

# CERTIFICATE OF ANALYSIS

**12X 352 (batch C)**

## Certified Reference Material Information

Type: LOW-ALLOY STEEL (WROUGHT)

Form and Size: Disc 42mm Diameter x 15mm Thickness

Manufactured by: Polycast Limited

Certified and Supplied by: MBH Analytical Limited

## Certified Analysis

### Percentage element by weight

Element	C	Si	S	P	Mn	Ni	Cr	Mo
Value <sup>1</sup>	0.257	0.498	0.131	0.074	0.533	0.380	0.348	0.242
Uncertainty <sup>2</sup>	0.005	0.010	0.005	0.003	0.011	0.007	0.005	0.006

Element	Cu	Co	Sn	Al	W	Ti	V	As
Value <sup>1</sup>	0.162	0.031	0.124	0.090	0.140	0.246	0.042	0.030
Uncertainty <sup>2</sup>	0.004	0.002	0.006	0.004	0.012	0.006	0.002	0.002

## Definitions

- <sup>1</sup> The assigned 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 20th March 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**

Milled samples for chemical analysis were taken from several positions within the batch. In addition, at least 10% of all discs were selected for homogeneity checking.

## **Homogeneity**

The discs were checked for lateral 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 mostly 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 grinding. 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.

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
1	0.242	0.479	0.121	0.0650	0.509	0.363	0.332	0.230
2	0.251	0.486	0.122	0.0668	0.509	0.368	0.333	0.230
3	0.254	0.487	0.124	0.0715	0.509	0.369	0.338	0.233
4	0.255	0.490	0.126	0.0720	0.516	0.370	0.344	0.238
5	0.256	0.491	0.126	0.0727	0.520	0.372	0.346	0.239
6	0.256	0.500	0.129	0.0736	0.536	0.381	0.348	0.240
7	0.257	0.504	0.132	0.0737	0.539	0.385	0.350	0.247
8	0.258	0.505	0.133	0.075	0.542	0.386	0.352	0.248
9	0.260	0.516	0.135	0.0792	0.547	0.387	0.353	0.249
10	0.262	0.519	0.138	0.0807	0.550	0.390	0.354	0.250
11	0.275		0.140	0.081	0.550	0.391	0.355	0.257
12			0.145	0.0810	0.550	0.392	0.355	
13					0.551		0.358	
Mean	0.257	0.498	0.131	0.074	0.533	0.380	0.348	0.242
Std Dev	0.008	0.013	0.008	0.005	0.018	0.010	0.009	0.009
C <sub>(95%)</sub>	0.005	0.010	0.005	0.003	0.011	0.007	0.005	0.006

Sample	Cu	Co	Sn	Al	W	Ti	V	As
1	0.153	0.0260	0.114	0.0813	0.120	0.230	0.0361	0.0265
2	0.157	0.0264	0.115	0.0830	0.124	0.240	0.0375	0.0269
3	0.158	0.0285	0.117	0.084	0.126	0.243	0.0401	0.0270
4	0.159	0.0307	0.120	0.0880	0.131	0.244	0.0409	0.0278
5	0.160	0.0317	0.125	0.0898	0.145	0.246	0.0421	0.028
6	0.161	0.0319	0.126	0.0909	0.150	0.248	0.0422	0.0290
7	0.161	0.032	0.127	0.0930	0.151	0.248	0.0422	0.030
8	0.163	0.032	0.129	0.0939	0.152	0.250	0.0425	0.0309
9	0.163	0.0322	0.133	0.0940	0.157	0.252	0.043	0.0324
10	0.165	0.0327	0.133	0.0986		0.252	0.0432	0.0325
11	0.168	0.0328				0.254	0.045	0.0327
12	0.168						0.046	0.0344
13	0.172						0.0461	
Mean	0.162	0.0306	0.124	0.090	0.140	0.246	0.0421	0.0298
Std Dev	0.005	0.0025	0.007	0.006	0.014	0.007	0.0029	0.0027
C <sub>(95%)</sub>	0.003	0.0017	0.005	0.004	0.011	0.005	0.0018	0.0017

Note: C<sub>(95%)</sub> 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  
Bodycote Materials Testing  
Sheffield Testing Laboratories Ltd  
Universal Scientific Laboratory  
Westmoreland Testing and Research, Inc  
Institute of Iron and Steel Technology  
Luo Yang Copper Co  
Quality Inspection Station, Wu Han Steel  
Sargam Metals Pvt Ltd  
TCR Engineering Services Ltd  
Laboratory TUV Nord Czech  
De Bruyn Spectroscopic Solutions Ltd  
Genitest Inc  
Colleshill Laboratories Ltd

Sheffield, England  
Middlesbrough, England  
Sheffield, England  
Milperra, NSW, Australia  
Youngstown, PA, USA  
Shanghai, China  
Luo Yang, He Nan, China  
Wu Han, Hubei, China  
Chennai, India  
Mumbai, India  
Brno, Czech Republic  
Johannesburg, South Africa  
Montreal, Canada  
Colleshill, England

UKAS accreditation 1385  
UKAS accreditation 0239  
UKAS accreditation 0136  
NATA accreditation 0492  
A2LA accreditation 0621  
CNAL accreditation 0783  
CNAL accreditation 0173  
CNAL accreditation 0271  
NABL accreditation 0025  
NABL accreditation 0367  
CAI accreditation 1060

Note: to achieve National Accreditation (eg UKAS, A2LA, 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	FAAS	OTHER
Carbon	-	-	1-11 combustion (infra-red detection)
Silicon	4, 5, 7	-	1, 3, 6, 9, 10 gravimetric (perchloric acid)
			2, 8 photometric (molybdenum blue)
Sulfur	5, 6	-	1, 2, 4, 7-12 combustion (infra-red detection)
			3 combustion (volumetric detection)
Phosphorus	2, 4, 8, 10-12	-	1, 3, 5, 7 photometric (molybdenum blue)
			6, 9 volumetric (alkalimetric)
Manganese	3-6, 10, 11, 13	1, 2	8, 12 volumetric (arsenite)
			7, 9 photometric (periodate)
Nickel	1-4, 8, 11	5, 6, 9, 12	7, 10 photometric (dimethyl glyoxime)
Chromium	1, 3, 7, 9-11, 13	2, 4, 5, 8	6, 12 volumetric (FAS)
Molybdenum	2, 3, 5, 8, 10, 11	6, 7	1, 4, 9 photometric (thiocyanate)
Copper	1-3, 5, 6, 10, 11, 13	7, 8, 9, 12	4 photometric (BCO)
Cobalt	4, 5, 7-11	1-3, 6	
Tin	1-4, 6, 8-10	5, 7	
Aluminium	3, 4, 6-10	2, 5	1 photometric (chrome azurol S)
Tungsten	1, 3, 5-9	-	4 volumetric (titanium chloride)
			2 photometric (thiocyanate); 3: ICP-MS
Titanium	2-5, 7, 9-11	6	8 photometric (DAP); 1: ICP-MS
Vanadium	3-5, 7-11, 13	6, 12	2 volumetric (FFAS); 1: ICP-MS
Arsenic	1-5, 8-10, 12	6, 7	11 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 ASTM E1724 and 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 March 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.