

# CERTIFICATE OF ANALYSIS

**12X LA4 (batch B)**

## 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
Value <sup>1</sup>	0.537	0.335	0.039	0.0363	0.303	0.521	0.499
Uncertainty <sup>2</sup>	0.010	0.005	0.003	0.0011	0.010	0.009	0.011

Element	Mo	Cu	Co	Al	W	V	N
Value <sup>1</sup>	0.489	0.334	0.105	0.057	0.091	0.328	0.0222
Uncertainty <sup>2</sup>	0.010	0.005	0.003	0.004	0.006	0.007	0.0019

## 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 16th April 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
1	0.520	0.321	0.0322	0.0346	0.277	0.498	0.474
2	0.521	0.326	0.0332	0.0350	0.278	0.500	0.474
3	0.523	0.331	0.0345	0.0353	0.288	0.506	0.481
4	0.526	0.332	0.038	0.0358	0.288	0.508	0.487
5	0.538	0.332	0.0381	0.0362	0.297	0.515	0.490
6	0.541	0.332	0.039	0.0363	0.303	0.515	0.503
7	0.543	0.335	0.0395	0.0366	0.310	0.525	0.505
8	0.544	0.336	0.0411	0.0376	0.310	0.530	0.508
9	0.545	0.340	0.0422	0.0394	0.310	0.531	0.510
10	0.545	0.342	0.0424		0.315	0.532	0.511
11	0.548	0.344	0.0434		0.318	0.535	0.512
12	0.550	0.350	0.0437		0.321	0.536	0.528
13					0.328	0.540	
<b>Mean</b>	<b>0.537</b>	<b>0.335</b>	<b>0.0389</b>	<b>0.0363</b>	<b>0.303</b>	<b>0.521</b>	<b>0.499</b>
<b>Std Dev</b>	0.011	0.008	0.0039	0.0015	0.016	0.015	0.017
<b>C<sub>(95%)</sub></b>	0.007	0.005	0.0025	0.0011	0.010	0.009	0.011

Sample	Mo	Cu	Co	Al	W	V	N
1	0.466	0.324	0.098	0.0458	0.0831	0.315	0.0174
2	0.470	0.324	0.099	0.0498	0.0850	0.315	0.0210
3	0.478	0.325	0.104	0.0532	0.086	0.316	0.0218
4	0.479	0.326	0.105	0.0535	0.0862	0.320	0.0225
5	0.481	0.328	0.105	0.054	0.0908	0.324	0.023
6	0.485	0.333	0.106	0.0553	0.0935	0.328	0.023
7	0.491	0.335	0.107	0.058	0.0982	0.330	0.024
8	0.497	0.335	0.108	0.0590	0.1042	0.331	0.025
9	0.503	0.340	0.108	0.060		0.336	
10	0.505	0.341	0.109	0.0601		0.339	
11	0.507	0.341	0.109	0.0641		0.341	
12	0.511	0.344		0.0651		0.345	
13		0.347		0.0667			
<b>Mean</b>	<b>0.489</b>	<b>0.334</b>	<b>0.105</b>	<b>0.057</b>	<b>0.091</b>	<b>0.328</b>	<b>0.0222</b>
<b>Std Dev</b>	0.015	0.008	0.004	0.006	0.007	0.011	0.0023
<b>C<sub>(95%)</sub></b>	0.010	0.005	0.003	0.004	0.006	0.007	0.0019

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	Sheffield, England	UKAS accreditation 1385
Bodycote Materials Testing	Middlesbrough, England	UKAS accreditation 0239
Universal Scientific Laboratory	Milperra, NSW, Australia	NATA accreditation 0492
Westmoreland Testing and Research, Inc	Youngstown, PA, USA	A2LA accreditation 0621
Institute of Iron and Steel Technology	Shanghai, China	CNAL accreditation 0783
Luo Yang Copper Co	Luo Yang, He Nan, China	CNAL accreditation 0173
Quality Inspection Station, Wu Han Steel	Wu Han, Hubei, China	CNAL accreditation 0271
Sargam Metals Pvt Ltd	Chennai, India	NABL accreditation 0025
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
Laboratory TUV Nord Czech	Brno, Czech Republic	CAI accreditation 1060
De Bruyn Spectroscopic Solutions Ltd	Johannesburg, South Africa	
Genitest Inc	Montreal, Canada	
Coleshill Laboratories Ltd	Coleshill, England	

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	2, 3, 5, 10, 12	-	12	combustion (volumetric detection)
			1, 6-8, 11	gravimetric (perchloric acid)
Sulfur	9, 12	-	4, 9	photometric (molybdenum blue)
			1-7, 10, 11	combustion (infra-red detection)
Phosphorus	1, 2, 3	-	8	combustion (volumetric detection)
			4, 5, 7, 8	photometric (molybdenum blue)
Manganese	2, 4, 6, 7, 10, 13	3, 5, 12	6, 9	volumetric (alkalimetric)
			1, 9, 11	volumetric (FAS, arsenite)
Nickel	1, 3, 4, 6, 8, 10, 12	2, 5, 7, 9	8	photometric (periodate)
			11, 13	gravimetric (dimethyl glyoxime)
Chromium	3, 5, 6, 9-12	1, 2, 7	4, 8	volumetric (ferrous ammonium sulfate)
Molybdenum	3, 4, 7, 9-12	2, 8	1, 5, 6	photometric (thiocyanate)
Copper	5, 7-11	1-4, 12, 13	6	photometric (BCO)
Cobalt	1-7, 9	8, 10, 11		
Aluminium	3-9, 11, 13	2, 10, 12	1	photometric (chrome azurol S)
Tungsten	1, 3, 5, 7, 8	-	2	volumetric (titanium chloride)
			4	photometric (thiocyanate); 6: ICP-MS
Vanadium	2-5, 8, 9, 11, 12	1, 6, 10	7	volumetric (ferrous ammonium sulfate)
Nitrogen	-	-	3, 4, 6, 7	inert gas fusion (thermal conductivity)
			1, 2, 5, 8	volumetric (hydrochloric acid, after distillation)

## 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 April 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.