

# CERTIFICATE OF ANALYSIS

**12X 15253 (batch S)**

## Certified Reference Material Information

Type: LOW-ALLOY STEEL (WROUGHT)  
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	Cu
Value <sup>1</sup>	0.173	0.299	0.053	0.0552	0.992	0.506	1.505	0.513	0.326
Uncertainty <sup>2</sup>	0.004	0.006	0.002	0.0012	0.015	0.006	0.008	0.007	0.004

Element	Co	Sn	Al	V	W	Nb	Ta	As	N
Value <sup>1</sup>	0.0898	0.129	0.024	0.242	0.265	0.383	0.011	0.0259	0.0235
Uncertainty <sup>2</sup>	0.0014	0.004	0.002	0.003	0.007	0.006	0.002	0.0010	0.0010

## 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 1<sup>st</sup> July 2014  
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 - 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**

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	Cu
1	0.167	0.285	0.0498	0.0519	0.951	0.495	1.481	0.496	0.315
2	0.167	0.286	0.0503	0.0535	0.951	0.495	1.487	0.497	0.319
3	0.170	0.286	0.0508	0.0546	0.962	0.497	1.493	0.499	0.320
4	0.172	0.292	0.0520	0.0548	0.979	0.499	1.496	0.504	0.320
5	0.172	0.295	0.0524	0.0549	0.981	0.501	1.499	0.507	0.324
6	0.172	0.299	0.0526	0.0549	0.985	0.504	1.499	0.511	0.324
7	0.173	0.301	0.0534	0.0549	0.987	0.505	1.500	0.513	0.327
8	0.176	0.302	0.0535	0.0552	0.992	0.508	1.501	0.520	0.328
9	0.177	0.304	0.0535	0.0556	0.999	0.509	1.511	0.522	0.328
10	0.179	0.309	0.0536	0.0570	1.007	0.512	1.512	0.523	0.329
11	0.180	0.310	0.0538	0.0570	1.010	0.512	1.514	0.525	0.331
12		0.313	0.0545	0.0585	1.025	0.514	1.518	0.526	0.336
13			0.0550		1.028	0.517	1.526	0.528	0.338
14			0.0551		1.033	0.519	1.527		
15							1.532		
<b>Mean</b>	<b>0.173</b>	<b>0.299</b>	<b>0.0529</b>	<b>0.0552</b>	<b>0.992</b>	<b>0.506</b>	<b>1.505</b>	<b>0.513</b>	<b>0.326</b>
<b>Std Dev</b>	0.004	0.010	0.0017	0.0017	0.026	0.008	0.015	0.012	0.007
<b>C<sub>(95%)</sub></b>	0.003	0.006	0.0010	0.0011	0.015	0.005	0.008	0.007	0.004

Sample	Co	Sn	Al	V	W	Nb	Ta	As	N
1	0.0866	0.124	0.0190	0.233	0.248	0.371	0.0088	0.0234	0.0209
2	0.0872	0.125	0.0199	0.235	0.252	0.372	0.0096	0.0245	0.0216
3	0.0887	0.126	0.0211	0.235	0.259	0.375	0.0103	0.0246	0.0225
4	0.0888	0.127	0.0213	0.235	0.262	0.377	0.0104	0.0248	0.0232
5	0.0893	0.128	0.0225	0.237	0.263	0.378	0.0124	0.0257	0.0235
6	0.0893	0.128	0.0228	0.239	0.265	0.381	0.0131	0.0258	0.0238
7	0.0900	0.129	0.0229	0.239	0.265	0.385	0.0150	0.0262	0.0244
8	0.0902	0.129	0.0243	0.241	0.271	0.387		0.0263	0.0247
9	0.0905	0.129	0.0246	0.242	0.273	0.388		0.0269	0.0251
10	0.0909	0.131	0.0248	0.243	0.276	0.389		0.0269	0.0252
11	0.0914	0.131	0.0252	0.244	0.280	0.389		0.0274	
12	0.0915	0.131	0.0275	0.248		0.390		0.0278	
13	0.0933	0.133	0.0281	0.249		0.396			
14		0.134	0.0286	0.250					
15		0.135	0.0287	0.250					
16				0.251					
<b>Mean</b>	<b>0.0898</b>	<b>0.129</b>	<b>0.0241</b>	<b>0.242</b>	<b>0.265</b>	<b>0.383</b>	<b>0.0114</b>	<b>0.0259</b>	<b>0.0235</b>
<b>Std Dev</b>	0.0018	0.003	0.0031	0.006	0.010	0.008	0.0022	0.0013	0.0015
<b>C<sub>(95%)</sub></b>	0.0011	0.002	0.0017	0.003	0.007	0.005	0.0020	0.0008	0.0010

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

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
Laboratory Testing, Inc	Hatfield, PA, USA	A2LA accreditation 0117
Shanghai Jinyi Test Tech Co	Shanghai, China	CNAS accreditation L0041
Shandong Metallurgical & Science Research	Jinan, Shandong, China	CNAS accreditation 1461
Bureau Veritas CPS Pvt	Chennai, India	NABL accreditation 0025
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	
Mineral & Metallurgical Laboratories	Bangalore, India	

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	1, 3, 5, 7, 8, 10	-	2, 9, 11 photometric (molybdenum blue)
Sulfur	7	-	4, 6, 12 gravimetric (perchloric acid)
Phosphorus	1-4, 6, 7, 10, 11	-	1-6, 8-14 combustion (IR or volumetric detection)
Manganese	1-3, 5, 7, 8, 11-13	6, 10	5, 9 photometric (molybdenum blue)
Nickel	1-5, 7, 8, 10, 11	9, 13	8, 12 volumetric (alkalimetric)
Chromium	1-4, 8-11, 13-15	7	4 photometric (periodate)
Molybdenum	1, 3, 4, 6, 7, 9, 10, 12	8, 13	9, 14 volumetric (arsenite)
Copper	1, 2, 4-6, 9-11, 13	3, 7, 8	6 gravimetric (dimethyl glyoxime)
Cobalt	1, 2, 5-8, 10-13	3, 9	12, 14 photometric (dimethyl glyoxime/EDTA)
Tin	3-6, 8-15	1, 2, 7	5, 6, 12 volumetric (ferrous ammonium sulfate)
Aluminium	2, 4, 6-8, 10, 11, 13-15	1, 3, 12	2, 5, 11 photometric (thiocyanate)
Vanadium	1, 3-7, 9-12	2, 8, 15	12 photometric (BCO)
Tungsten	1-5, 9-11	6	4 volumetric (iodine)
Niobium	1, 3-5, 7, 8, 10-12	2	5, 9 photometric (chrome azurol S)
Tantalum	2-7	-	7 volumetric (ferrous ammonium sulfate)
Arsenic	1-3, 5, 6, 8-12	4, 7	7 photometric (thiocyanate)
Nitrogen	-	-	8 ICP-MS
			6 photometric (chlorosulfophenol)
			9 ICP-MS
			13 gravimetric (cupferron)
			1 ICP-MS
			1, 4-6, 9, 10 inert gas fusion (thermal conductivity)
			2, 3, 7 volumetric (hydrochloric acid)
			8 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).

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 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 July 2034, 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.