

# CERTIFICATE OF ANALYSIS

**13X 34700 (batch A)**

## Certified Reference Material Information

Type: STAINLESS STEEL (WROUGHT)  
Form and Size: Disc, ~38mm diameter  
Manufactured by: Stock Bar  
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>	0.016	0.480	(0.0005)	0.0276	1.283	9.32	17.22	0.392	0.165
Uncertainty <sup>2</sup>	0.002	0.006	-	0.0009	0.014	0.06	0.07	0.006	0.003

Element	Co	V	Nb	W	Al	Pb	Sn	B	N
Value <sup>1</sup>	0.132	0.123	0.329	0.144	0.025	(0.003)	0.0053	0.0007	0.0163
Uncertainty <sup>2</sup>	0.003	0.004	0.005	0.006	0.005	-	0.0008	0.0001	0.0009

Note: values given in parentheses are not certified - they are provided for information only.

## 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 28th February 2012



## **Method of Preparation**

This reference material was produced from commercial barstock to UNS S34700. The bar was prepared by electric arc melting & electroslag refining, followed by hot forging, soak and quench.

## **Sampling**

Samples for chemical analysis were taken from various positions throughout the batch. At least 10% of all discs were selected for non-destructive homogeneity testing.

## **Homogeneity**

The discs were checked for lateral segregation, and for local and batch homogeneity 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	Cu
1	0.0120	0.467	0.0001	0.0262	1.245	9.197	17.12	0.378	0.156
2	0.0130	0.471	0.0002	0.0268	1.250	9.221	17.18	0.383	0.159
3	0.0135	0.474	0.0004	0.0268	1.267	9.241	17.18	0.383	0.160
4	0.0138	0.478	0.0005	0.0270	1.276	9.253	17.18	0.385	0.161
5	0.0140	0.479	0.0008	0.0273	1.281	9.280	17.19	0.389	0.161
6	0.0151	0.484	0.0008	0.0275	1.284	9.286	17.20	0.392	0.163
7	0.0163	0.485	0.0009	0.0275	1.290	9.366	17.20	0.393	0.164
8	0.0166	0.485	<0.001	0.0277	1.292	9.380	17.20	0.395	0.166
9	0.0180	0.488	<0.001	0.0278	1.295	9.381	17.31	0.395	0.167
10	0.0184	0.491		0.0282	1.299	9.410	17.33	0.396	0.167
11	0.0190			0.0291	1.308	9.433	17.33	0.401	0.170
12	0.0190			0.0298	1.314	9.434		0.402	0.170
13	0.0207							0.402	0.170
14									0.171
<b>Mean</b>	<b>0.0161</b>	<b>0.480</b>	<b>0.0005</b>	<b>0.0276</b>	<b>1.283</b>	<b>9.324</b>	<b>17.22</b>	<b>0.392</b>	<b>0.165</b>
<b>Std Dev</b>	0.0028	0.008	0.0003	0.0010	0.021	0.086	0.07	0.008	0.005
<b>C (95%)</b>	0.0017	0.006	0.0003	0.0006	0.013	0.055	0.05	0.005	0.003

Sample	Co	V	Nb	W	Al	Pb	Sn	B	N
1	0.124	0.118	0.318	0.131	0.0201	0.0013	0.0028	0.0005	0.0135
2	0.124	0.120	0.320	0.135	0.0211	0.0020	0.0040	0.0006	0.0149
3	0.126	0.120	0.321	0.135	0.0222	0.0020	0.0050	0.0006	0.0154
4	0.128	0.121	0.324	0.136	0.0224	0.0043	0.0050	0.0007	0.0160
5	0.130	0.122	0.326	0.144	0.0226	0.0048	0.0051	0.0007	0.0164
6	0.131	0.122	0.327	0.145	0.0228	0.0057	0.0051	0.0008	0.0166
7	0.133	0.128	0.329	0.149	0.0228		0.0056	0.0008	0.0167
8	0.134	0.130	0.331	0.151	0.0264		0.0060	0.0008	0.0168
9	0.134	0.130	0.332	0.152	0.0271		0.0061	0.0008	0.0170
10	0.135		0.333	0.152	0.0280		0.0064	0.0010	0.0174
11	0.135		0.335	0.156	0.0281		0.0075		0.0187
12	0.137		0.340		0.0289				
13	0.138		0.344		0.0290				
14	0.141								
<b>Mean</b>	<b>0.132</b>	<b>0.123</b>	<b>0.329</b>	<b>0.144</b>	<b>0.0247</b>	<b>(0.0034)</b>	<b>0.0053</b>	<b>0.0007</b>	<b>0.0163</b>
<b>Std Dev</b>	0.005	0.005	0.008	0.009	0.0032	-	0.0012	0.0001	0.0014
<b>C (95%)</b>	0.003	0.004	0.005	0.006	0.0019	-	0.0008	0.0001	0.0009

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 Materials Testing  
Metals Technology (Testing) Ltd  
Sheffield Assay Office  
Universal Scientific Laboratory  
Laboratory Testing, Inc  
Genitest, Inc  
Institute of Iron and Steel Technology  
Luo Yang Copper Co  
Sargam Metals Pvt Ltd  
TCR Engineering Services Ltd  
Raghavendra Spectrometallurgical Lab.  
De Bruyn Spectroscopic Solutions Ltd  
Coleshill Laboratories Ltd  
London & Scandinavian Met. Co Ltd  
LECO Corporation

Middlesbrough, England  
Sheffield, England  
Sheffield, England  
Milperra, NSW, Australia  
Hatfield, PA, USA  
Montreal, Canada  
Shanghai, China  
Luo Yang, He Nan, China  
Chennai, India  
Mumbai, India  
Bangalore, India  
Johannesburg, South Africa  
Birmingham, England  
Rotherham, England  
St Joseph, MI, USA

UKAS accreditation 0239  
UKAS accreditation 0963  
UKAS accreditation 0012  
NATA accreditation 0492  
A2LA accreditation 0117  
PRI accreditation 123077  
CNAL accreditation 0783  
CNAL accreditation 0173  
NABL accreditation 0025  
NABL accreditation 0367  
NABL accreditation 0371

Note: to achieve the above-noted accreditation (eg UKAS, NATA, etc), test houses must demonstrate conformity to the general requirements of EN ISO/IEC 17025.

## Analytical Methods Used

ELEMENT	RESULT No. & METHOD				
	ICP-AES	FAAS	GD-AES	OTHER	
Carbon	-	-	-	all	combustion (IR or volumetric detection)
Silicon	1-3, 8, 9	-	-	5, 7, 10	photometric (molybdenum blue)
Sulfur	2	-	-	4, 6	gravimetric (perchloric acid)
Phosphorus	1, 3, 9-12	-	4	1, 3-9	combustion (IR or volumetric detection)
Manganese	1, 2, 6, 9, 11, 12	7	10	2, 7	volumetric (alkalimetric)
Nickel	1, 2, 4, 6, 10, 11	9	3	5, 6, 8	photometric (molybdenum blue)
Chromium	2, 3, 5, 8, 9	-	11	3, 4	photometric (periodate)
Molybdenum	1, 3, 4, 6, 10-13	2	9	5, 8	volumetric (ferrous ammonium sulfate)
Copper	1, 2, 5, 7, 9-12, 14	6, 8	13	7, 12	gravimetric (dimethyl glyoxime)
Cobalt	1-3, 5-7, 10-13	4, 8	9	1, 4, 6, 7, 10	volumetric (dimethyl glyoxime/EDTA)
Vanadium	1, 3, 6-8	2, 5, 9	-	5	volumetric (ferrous ammonium sulfate)
Niobium	1-6, 8, 12, 13	9	-	7, 8	ICP-MS
Tungsten	2-6, 9-11	-	8	3	photometric (thiocyanate)
Aluminium	3, 6, 7, 9, 11-13	4, 5, 8	10	4	volumetric (thiosulfate)
Lead	1, 4, 6	2, 3, 5	-	14	photometric (BCO)
Tin	1-3, 6, 8-11	4, 5, 7	-	4	photometric (5-chloro PADAB)
Boron	1, 3, 5-10	-	2	7	volumetric (ferrous ammonium sulfate)
Nitrogen	-	-	-	10, 11	ICP-MS
				1	photometric (4-PAR)
				7	gravimetric (cinchonine)
				1, 2	ICP-MS
					photometric (chrome azurol S)
				4	ICP-MS
				6, 8	volumetric (hydrochloric acid)
				1-5, 7, 9-11	inert gas fusion (thermal conductivity)

## Notes

This Certified Reference Material has been produced and certified 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 OES, it is appropriate to avoid usage of the central portion of approx 6mm 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. This certification will therefore expire in February 2032, 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.