

# CERTIFICATE OF ANALYSIS

**13X 64152 (batch A)**

## Certified Reference Material Information

Type: HIGH STRENGTH STAINLESS STEEL (WROUGHT)

Form and Size: Disc, ~38mm diameter

Manufactured by: Tata Steel UK Ltd

Certified and Supplied by: MBH Analytical Ltd

## Assigned Values

### Percentage element by weight

Element	C	Si	S	P	Mn	Ni	Cr
Value <sup>1</sup>	0.114	0.224	0.0020	0.0123	0.666	2.50	11.34
Uncertainty <sup>2</sup>	0.003	0.005	0.0002	0.0006	0.006	0.02	0.05

Element	Mo	Cu	Co	V	Al	Sn	N
Value <sup>1</sup>	1.567	0.0622	0.0185	0.275	0.0315	0.0053	0.0339
Uncertainty <sup>2</sup>	0.013	0.0012	0.0007	0.003	0.0013	0.0005	0.0009

## 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 21st July 2015

C Eveleigh



## **Method of Preparation**

This reference material was produced from a single length of stock commercial bar with chemistry to UNS S64152, also known as 'Jethete' and 'M152'. The steel was arc-furnace melted and vacuum degassed, prior to ingot casting; the subsequent metallurgical history is unknown.

## **Sampling**

Samples for chemical analysis were taken from various positions throughout the batch. Approximately 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 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**

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 milling, turning or 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	0.109	0.212	0.0015	0.0110	0.651	2.463	11.25
2	0.111	0.215	0.0015	0.0112	0.654	2.465	11.30
3	0.112	0.217	0.0019	0.0115	0.657	2.467	11.30
4	0.112	0.221	0.0020	0.0120	0.659	2.470	11.31
5	0.112	0.223	0.0020	0.0121	0.660	2.472	11.31
6	0.113	0.225	0.0020	0.0123	0.664	2.483	11.32
7	0.113	0.228	0.0020	0.0124	0.665	2.490	11.35
8	0.113	0.228	0.0020	0.0125	0.666	2.493	11.35
9	0.113	0.229	0.0021	0.0127	0.668	2.494	11.39
10	0.117	0.233	0.0021	0.0130	0.671	2.511	11.40
11	0.118	0.235	0.0022	0.0136	0.676	2.530	11.43
12	0.120		0.0023	0.0138	0.681	2.543	
13					0.688	2.558	
<b>Mean</b>	<b>0.114</b>	<b>0.224</b>	<b>0.0020</b>	<b>0.0123</b>	<b>0.666</b>	<b>2.495</b>	<b>11.34</b>
<b>Std Dev</b>	0.003	0.007	0.0002	0.0009	0.011	0.031	0.05
<b>C<sub>(95%)</sub></b>	0.002	0.005	0.0002	0.0006	0.006	0.019	0.04

Sample	Mo	Cu	Co	V	Al	Sn	N
1	1.530	0.0598	0.0162	0.265	0.0279	0.0040	0.0321
2	1.538	0.0599	0.0165	0.268	0.0284	0.0044	0.0327
3	1.551	0.0600	0.0168	0.270	0.0299	0.0049	0.0334
4	1.551	0.0608	0.0173	0.273	0.0319	0.0050	0.0335
5	1.565	0.0615	0.0180	0.274	0.0319	0.0052	0.0336
6	1.569	0.0617	0.0180	0.274	0.0325	0.0052	0.0344
7	1.571	0.0625	0.0182	0.275	0.0327	0.0054	0.0346
8	1.573	0.0629	0.0190	0.275	0.0327	0.0055	0.0351
9	1.588	0.0632	0.0193	0.277	0.0329	0.0055	0.0355
10	1.590	0.0635	0.0193	0.277	0.0329	0.0058	
11	1.591	0.0641	0.0194	0.278	0.0329	0.0059	
12	1.592	0.0642	0.0195	0.282		0.0062	
13		0.0644	0.0196	0.284			
14			0.0198				
15			0.0206				
<b>Mean</b>	<b>1.567</b>	<b>0.0622</b>	<b>0.0185</b>	<b>0.275</b>	<b>0.0315</b>	<b>0.0053</b>	<b>0.0339</b>
<b>Std Dev</b>	0.021	0.0017	0.0013	0.005	0.0019	0.0006	0.0011
<b>C<sub>(95%)</sub></b>	0.013	0.0010	0.0007	0.003	0.0013	0.0004	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 Ltd	Middlesbrough, England	UKAS accreditation 0239
Sheffield Assay Office	Sheffield, England	UKAS accreditation 0012
Birmingham Assay Office	Birmingham, England	UKAS accreditation 0667
Metals Technology (Testing) Ltd	Sheffield, England	UKAS accreditation 0963
Universal Scientific Laboratory	Milperra, NSW, Australia	NATA accreditation 0492
Genitest, Inc	Montreal, Canada	PRI accreditation 123077
Shanghai Jinyi Test Technology Co	Shanghai, China	CNAL accreditation 0783
Shandong Metallurgical & Science Research	Jinan, Shandong, China	CNAS accreditation 1461
Bureau Veritas CPS Pvt Ltd	Chennai, India	NABL accreditation 0025
TCR Engineering Services Ltd	Mumbai, India	NABL accreditation 0367
Raghavendra Spectrometallurgical Lab.	Bangalore, India	NABL accreditation 0371
Instytut Metalurgii Zelaza	Gliwice, Poland	PCA accreditation AB554
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
London & Scandinavian Met Co Ltd	Rotherham, England	
Coleshill Laboratories Ltd	Birmingham, England	
Analyticka Laborator Lithea sro	Brno, Czech Republic	

Note: to achieve the above accreditation (eg 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, 2, 4, 5, 8, 9	-	3, 7, 11 photometric (molybdenum blue) 6, 10 gravimetric (perchloric acid)
Sulfur	9	-	1-8, 10-12 combustion (IR or volumetric detection)
Phosphorus	1-3, 6-9, 12	-	4, 5, 10 photometric (molybdenum blue) 11 volumetric (alkalimetric)
Manganese	1-4, 6-8, 13	5, 10	9, 11 photometric (periodate) 12 volumetric (arsenite)
Nickel	1, 3, 6, 8, 9, 11-13	5	2, 10 photometric (diethyl glyoxime) 4, 7 gravimetric (dimethyl glyoxime)
Chromium	1, 5, 6, 8	-	2-4, 7, 9-11 volumetric (ferrous ammonium sulfate)
Molybdenum	1, 3, 6-10, 12	2, 11	4, 5 photometric (thiocyanate)
Copper	1-4, 6, 7, 9-11	8, 12	5, 13 photometric (BCO)
Cobalt	1-6, 10, 11, 13, 15	7, 12, 14	8 photometric (2 $\beta$ -naphthol) 9 volumetric (iodine)
Vanadium	1, 3-5, 7-9, 11-13	2, 10	6 volumetric (ferrous ammonium sulfate)
Aluminium	2-4, 6, 8-10	1, 11	5, 7 photometric (chrome azurol S)
Tin	1-4, 6, 8-12	5, 7	
Nitrogen	-	-	1, 2, 4-6 inert gas fusion (thermal conductivity) 3, 9 volumetric (hydrochloric acid) 7, 8 photometric (Nessler 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 central portion, ~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 2035, although we reserve the right to make changes as issue revisions, in the intervening period.

The testing, 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.