

# CERTIFICATE OF ANALYSIS

## 13X NSA7 (batch B)

### Certified Reference Material Information

Type: NITROGEN STAINLESS STEEL (WROUGHT)  
Form and Size: Disc, ~41mm 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>	0.0130	0.278	0.0005	0.0160	0.864	6.37	25.69	3.28	1.53
Uncertainty <sup>2</sup>	0.0010	0.007	0.0002	0.0007	0.008	0.03	0.07	0.02	0.02

Element	Co	V	Nb	W	Al	Sn	Pb	B	N
Value <sup>1</sup>	0.0471	0.0802	(0.009)	0.133	0.0142	0.0020	0.0009	0.0018	0.232
Uncertainty <sup>2</sup>	0.0010	0.0014	-	0.004	0.0009	0.0002	0.0003	0.0003	0.005

Note: values 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 \_\_\_\_\_

on 4th July 2018

C Eveleigh

## **Method of Preparation**

This reference material was produced from commercial barstock type Ferralium 255 to UNS S32550. The bar was prepared by electric arc melting and argon-oxygen decarburization, followed by hot rolling. It was solution-treated, quenched and tempered then machined to size.

## **Sampling**

Samples for chemical analysis were taken from various positions throughout the batch. Approximately 5% 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, 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.

Of the individual results herein, some have traceability (to the mole) via primary analytical methods. Some are traceable to substances of known stoichiometry. Most have traceability via commercial solutions. Furthermore, some results have additional 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	0.0095	0.2580	0.00020	0.0141	0.8508	6.273	25.511	3.217	1.482
2	0.0105	0.2636	0.00038	0.0144	0.8540	6.282	25.521	3.227	1.491
3	0.0113	0.2693	0.00040	0.0148	0.8550	6.314	25.540	3.227	1.499
4	0.0122	0.2720	0.00040	0.0153	0.8590	6.320	25.550	3.245	1.504
5	0.0122	0.2737	0.00042	0.0154	0.8590	6.329	25.570	3.250	1.512
6	0.0130	0.2771	0.00050	0.0160	0.8606	6.358	25.600	3.263	1.520
7	0.0131	0.2812	0.00053	0.0162	0.8611	6.361	25.636	3.268	1.525
8	0.0135	0.2860	0.00070	0.0166	0.8696	6.361	25.654	3.277	1.528
9	0.0138	0.2877	0.00070	0.0166	0.8717	6.381	25.720	3.281	1.538
10	0.0142	0.2880	0.00080	0.0168	0.8792	6.386	25.723	3.289	1.556
11	0.0144	0.2904	0.00084	0.0168	0.8820	6.411	25.740	3.296	1.557
12	0.0144	0.2914		0.0173		6.419	25.800	3.312	1.557
13	0.0145			0.0183		6.423	25.838	3.326	1.572
14	0.0160					6.425	25.850	3.333	1.576
15						6.441	25.880	3.366	
16						6.452	25.888		
<b>Mean</b>	<b>0.0130</b>	<b>0.2782</b>	<b>0.00053</b>	<b>0.0160</b>	<b>0.8638</b>	<b>6.371</b>	<b>25.689</b>	<b>3.282</b>	<b>1.530</b>
<b>Std Dev</b>	0.0018	0.0110	0.00020	0.0012	0.0103	0.056	0.134	0.042	0.030
<b>C (95%)</b>	0.0010	0.0070	0.00014	0.0007	0.0070	0.030	0.071	0.023	0.018

Sample	Co	V	Nb	W	Al	Sn	Pb	B	N
1	0.0443	0.0769	0.0055	0.1233	0.0122	0.0014	0.0005	0.0012	0.2200
2	0.0451	0.0773	0.0071	0.1252	0.0128	0.0016	0.0005	0.0015	0.2220
3	0.0452	0.0777	0.0072	0.1256	0.0133	0.0018	0.0005	0.0016	0.2230
4	0.0460	0.0790	0.0074	0.1270	0.0138	0.0020	0.0007	0.0018	0.2267
5	0.0461	0.0793	0.0078	0.1277	0.0139	0.0021	0.0012	0.0018	0.2288
6	0.0466	0.0802	0.0078	0.1330	0.0143	0.0021	0.0012	0.0020	0.2310
7	0.0472	0.0810	0.0107	0.1350	0.0148	0.0021	0.0014	0.0020	0.2332
8	0.0474	0.0815	0.0115	0.1351	0.0153	0.0022	0.0014	0.0022	0.2367
9	0.0475	0.0822	0.0118	0.1370	0.0154	0.0022		0.0022	0.2391
10	0.0477	0.0825	0.0132	0.1370	0.0159	0.0022			0.2400
11	0.0477	0.0826		0.1394		0.0022			0.2410
12	0.0489	0.0827		0.1425					0.2439
13	0.0494			0.1430					
14	0.0498								
<b>Mean</b>	<b>0.0471</b>	<b>0.0802</b>	<b>0.0090</b>	<b>0.1331</b>	<b>0.0142</b>	<b>0.0020</b>	<b>0.0009</b>	<b>0.0018</b>	<b>0.2321</b>
<b>Std Dev</b>	0.0016	0.0022	0.0026	0.0067	0.0012	0.0003	0.0004	0.0003	0.0081
<b>C (95%)</b>	0.0009	0.0014	0.0018	0.0041	0.0009	0.0002	0.0003	0.0003	0.0052

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
Anchorcert Analytical	Birmingham, England	UKAS accreditation 0667
Metals Technology (Testing) Ltd	Sheffield, England	UKAS accreditation 0963
Universal Scientific Laboratory Pty Ltd	Sydney, 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	Shandong, Jinan, China	CNAS accreditation 1461
Raghavendra SpectroMet Laboratory	Bangalore, India	NABL accreditation T371
Genitest Inc	Montreal, Canada	PJ accreditation L17-153
Tec-Eurolab	Campogalliano, Italy	ACCREDIA accreditation 52
Instytut Metalurgii Zelaza	Gliwice, Poland	PCA accreditation AB554
TUV Nord Czech	Brno, Czech Republic	CAI accreditation L-1060
Mineral & Metallurgical Laboratories	Bangalore, India	
Analyticka Laborator Lithea sro	Brno, Czech Republic	
INCDMNR-IMNR	Pantelimon, Romania	
AMG Superalloys UK Ltd	Rotherham, England	

Note: to achieve the above accreditation (UKAS, 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, 6, 9, 11	-	4, 7, 12 gravimetric (perchloric acid)
Sulfur	11	-	8, 10 photometric (molybdenum blue)
Phosphorus	3, 5, 6, 8-13	-	1-10 combustion (IR or volumetric detection)
Manganese	1, 2, 5, 6, 8-10	3, 7	1, 4, 7 photometric (molybdenum blue)
Nickel	1-3, 5-7, 12, 14, 16	9, 10	2 volumetric (alkalimetric)
Chromium	3, 5, 6, 9, 10, 13, 15, 16	4	4 volumetric (arsenite)
Molybdenum	2-4, 7, 8, 10-13	5, 9	11 photometric (periodate)
Copper	2-4, 7, 8, 10-12, 14	1, 6, 9	4, 8, 11 gravimetric (dimethyl glyoxime)
Cobalt	1-3, 7, 9, 11-13	4, 5, 14	13, 15 photometric (dimethyl glyoxime)
Vanadium	1-6, 8, 10-12	7	1, 2, 7, 8, 11, 12, 14 volumetric (ferrous ammonium sulfate)
Niobium	1-4, 6-10	5	1, 6 photometric (thiocyanate)
Tungsten	1-3, 5, 8-12	13	14 gravimetric
Aluminium	1, 2, 4, 5, 7, 9	6, 10	5 volumetric (thiosulfate)
Tin	1, 3-11	2	13 photometric (BCO)
Lead	3, 5-8	1, 2, 4	6, 10 photometric (2 $\beta$ -naphthol)
Boron	1-9	-	8 volumetric (iodine)
Nitrogen	-	-	9 volumetric (ferrous ammonium sulfate)
			4, 7 photometric (thiocyanate)
			6 gravimetric
			3 volumetric (EDTA)
			8 photometric (chrome azurol S)
			1-4, 7-10 inert gas fusion (thermal conductivity)
			5, 6, 11 photometric (Nessler reagent)

## Notes

This Certified Reference Material has been produced and certified, wherever possible, in accordance with the requirements of ISO Guide 34, ISO Guide 31 and ISO Guide 35, 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. Technical support for this certification will therefore expire in July 2038, although we reserve the right to make changes as issue revisions, in the intervening period.

The procurement, 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.