

# CERTIFICATE OF ANALYSIS

## 13X NSA12 (batch A)

### Certified Reference Material Information

|                            |                                    |
|----------------------------|------------------------------------|
| Type:                      | NITROGEN STEEL TYPE 904L (WROUGHT) |
| Form and Size:             | Disc, 40mm diameter                |
| Manufactured by:           | Acciaierie Valbruna SPA, Italy     |
| Certified and Supplied by: | MBH Analytical Ltd                 |

### Assigned Values

#### Percentage element by weight

| Element                  | C      | Si    | S      | P      | Mn    | Ni    | Cr    | Mo   |
|--------------------------|--------|-------|--------|--------|-------|-------|-------|------|
| Value <sup>1</sup>       | 0.0192 | 0.492 | 0.0007 | 0.0267 | 1.272 | 24.84 | 19.63 | 4.20 |
| Uncertainty <sup>2</sup> | 0.0010 | 0.012 | 0.0002 | 0.0010 | 0.010 | 0.09  | 0.03  | 0.03 |

| Element                  | Cu    | Co    | V      | Nb     | W     | Al     | B      | N      |
|--------------------------|-------|-------|--------|--------|-------|--------|--------|--------|
| Value <sup>1</sup>       | 1.485 | 0.090 | 0.0660 | 0.0088 | 0.047 | 0.0169 | 0.0020 | 0.0662 |
| Uncertainty <sup>2</sup> | 0.014 | 0.003 | 0.0014 | 0.0007 | 0.003 | 0.0007 | 0.0002 | 0.0012 |

### 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 3<sup>rd</sup> August 2015

C Eveleigh



## **Method of Preparation**

This reference material was produced from commercial barstock, type 904L to UNS N08904. The bar was prepared by electric arc melting, argon-oxygen decarburization and continuous casting, followed by cold finishing and annealing.

## **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           |
|----------------|---------------|--------------|---------------|---------------|--------------|--------------|--------------|--------------|
| 1              | 0.0170        | 0.473        | 0.0003        | 0.0242        | 1.245        | 24.69        | 19.57        | 4.130        |
| 2              | 0.0180        | 0.475        | 0.0004        | 0.0245        | 1.248        | 24.77        | 19.59        | 4.143        |
| 3              | 0.0182        | 0.479        | 0.0005        | 0.0246        | 1.261        | 24.78        | 19.60        | 4.165        |
| 4              | 0.0183        | 0.489        | 0.0006        | 0.0252        | 1.261        | 24.80        | 19.62        | 4.168        |
| 5              | 0.0185        | 0.489        | 0.0006        | 0.0262        | 1.262        | 24.81        | 19.63        | 4.180        |
| 6              | 0.0187        | 0.491        | 0.0007        | 0.0268        | 1.274        | 24.85        | 19.64        | 4.183        |
| 7              | 0.0187        | 0.498        | 0.0009        | 0.0268        | 1.277        | 24.87        | 19.65        | 4.183        |
| 8              | 0.0190        | 0.505        | 0.0010        | 0.0268        | 1.279        | 24.92        | 19.65        | 4.189        |
| 9              | 0.0192        | 0.507        | 0.0010        | 0.0271        | 1.281        | 24.95        | 19.65        | 4.200        |
| 10             | 0.0199        | 0.512        | 0.0012        | 0.0279        | 1.284        | 24.99        | 19.66        | 4.202        |
| 11             | 0.0206        |              |               | 0.0281        | 1.294        |              | 19.66        | 4.250        |
| 12             | 0.0215        |              |               | 0.0285        | 1.295        |              | 19.66        | 4.251        |
| 13             | 0.0220        |              |               | 0.0299        |              |              |              | 4.271        |
| 14             |               |              |               |               |              |              |              | 4.285        |
| <b>Mean</b>    | <b>0.0192</b> | <b>0.492</b> | <b>0.0007</b> | <b>0.0267</b> | <b>1.272</b> | <b>24.84</b> | <b>19.63</b> | <b>4.200</b> |
| <b>Std Dev</b> | 0.0014        | 0.014        | 0.0003        | 0.0017        | 0.016        | 0.09         | 0.03         | 0.047        |
| <b>C (95%)</b> | 0.0009        | 0.010        | 0.0002        | 0.0010        | 0.010        | 0.07         | 0.02         | 0.027        |

| Sample         | Cu           | Co            | V             | Nb            | W             | Al            | B             | N             |
|----------------|--------------|---------------|---------------|---------------|---------------|---------------|---------------|---------------|
| 1              | 1.453        | 0.0846        | 0.0631        | 0.0071        | 0.0422        | 0.0153        | 0.0018        | 0.0639        |
| 2              | 1.454        | 0.0852        | 0.0633        | 0.0072        | 0.0431        | 0.0154        | 0.0018        | 0.0641        |
| 3              | 1.462        | 0.0872        | 0.0639        | 0.0077        | 0.0432        | 0.0159        | 0.0018        | 0.0652        |
| 4              | 1.465        | 0.0876        | 0.0644        | 0.0082        | 0.0436        | 0.0161        | 0.0019        | 0.0655        |
| 5              | 1.473        | 0.0894        | 0.0648        | 0.0086        | 0.0468        | 0.0164        | 0.0019        | 0.0658        |
| 6              | 1.475        | 0.0901        | 0.0650        | 0.0087        | 0.0470        | 0.0171        | 0.0020        | 0.0659        |
| 7              | 1.478        | 0.0905        | 0.0662        | 0.0091        | 0.0474        | 0.0171        | 0.0021        | 0.0664        |
| 8              | 1.483        | 0.0911        | 0.0667        | 0.0095        | 0.0480        | 0.0174        | 0.0021        | 0.0673        |
| 9              | 1.484        | 0.0920        | 0.0679        | 0.0098        | 0.0485        | 0.0175        | 0.0024        | 0.0688        |
| 10             | 1.500        | 0.0925        | 0.0685        | 0.0098        | 0.0492        | 0.0182        | 0.0024        | 0.0690        |
| 11             | 1.513        | 0.0949        | 0.0689        | 0.0101        | 0.0503        | 0.0182        |               |               |
| 12             | 1.513        |               | 0.0694        | 0.0103        | 0.0505        | 0.0182        |               |               |
| 13             | 1.515        |               |               |               | 0.0520        |               |               |               |
| 14             | 1.518        |               |               |               |               |               |               |               |
| <b>Mean</b>    | <b>1.485</b> | <b>0.0896</b> | <b>0.0660</b> | <b>0.0088</b> | <b>0.0471</b> | <b>0.0169</b> | <b>0.0020</b> | <b>0.0662</b> |
| <b>Std Dev</b> | 0.023        | 0.0032        | 0.0022        | 0.0011        | 0.0032        | 0.0011        | 0.0002        | 0.0017        |
| <b>C (95%)</b> | 0.013        | 0.0021        | 0.0014        | 0.0007        | 0.0019        | 0.0007        | 0.0002        | 0.0012        |

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 Pty Ltd   | Revesby, Australia     | NATA accreditation 492    |
| 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   |
| AMG Superalloys UK Ltd                    | Rotherham, England     |                           |
| Coleshill Laboratories Ltd                | Birmingham, England    |                           |
| Analyticka Laborator Lithea sro           | Brno, Czech Republic   |                           |

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, 8, 10               | -         | 4-6 gravimetric (perchloric acid)<br>7, 9 photometric (molybdenum blue)  |
| Sulfur     | 7                        | -         | 1-6, 8-10 combustion (IR or volumetric detection)  |
| Phosphorus | 1, 3, 4, 6, 7, 9, 12, 13 | -         | 2, 11 volumetric (alkalimetric)<br>5, 8, 10 photometric (molybdenum blue)  |
| Manganese  | 2, 6-10, 12              | 3, 4      | 1 volumetric (arsenite)<br>5, 11 photometric (periodate)   |
| Nickel     | 2, 4, 7                  | -         | 1, 3, 5, 9, 10 gravimetric (dimethyl glyoxime)<br>6, 8 photometric (dimethyl glyoxime)   |
| Chromium   | 1, 3, 5, 7, 11, 12       | -         | 2, 4, 6, 8-10 volumetric (ferrous ammonium sulfate)  |
| Molybdenum | 1-4, 6-8, 11, 12, 14     | 5, 9      | 10, 13 photometric (thiocyanate)   |
| Copper     | 1, 2, 4-6, 8, 12-14      | 7, 10, 11 | 3, 9 photometric (BCO)   |
| Cobalt     | 1, 3, 5, 6, 10, 11       | 2, 7, 8   | 4 photometric (2 $\beta$ -naphthol)<br>9 volumetric (iodine)   |
| Vanadium   | 1, 3-7, 10-12            | 2, 8, 9   |  |
| Niobium    | 1-3, 5, 7-10, 12         | 4, 11     | 6 photometric (chlorosulfophenol)  |
| Tungsten   | 1, 3-7, 9-11, 13         | 2, 8      | 12 photometric (thiocyanate)   |
| Aluminium  | 1-4, 6, 8, 10, 11        | 7, 9, 12  | 5 photometric (chrome azurol S)  |
| Boron      | 1-5, 7-10                | 6         |  |
| Nitrogen   |                          | -         | 1-3, 5, 8, 10 inert gas fusion (thermal conductivity)<br>4, 7, 9 volumetric (hydrochloric acid)<br>6 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 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 August 2035, although we reserve the right to make changes as issue revisions, in the intervening period.

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.