MBH ANALYTICAL LTD

HOLLAND HOUSE • QUEENS ROAD • BARNET • EN5 4DJ • ENGLAND • TEL: +44 (0)20 8441 2024 • FAX: +44 (0)20 8449 0810
email: info@mbh.co.uk web: www.mbh.co.uk

# CERTIFICATE OF ANALYSIS

## 13X 14219 (batch K)

## Certified Reference Material Information

Type: SPECIAL STAINLESS STEEL (CONTINUOUS CAST)

Form and Size: Disc ~40mm diameter

Manufactured by: Shenzhen Mingzhenwei Technology, China

Certified and Supplied by: MBH Analytical Ltd

## Assigned Values

**Percentage element by weight**

| Element | C | Si | S | P | Mn | Ni | Cr |
|---|---|---|---|---|---|---|---|
| **Value** [1] | **0.0997** | **1.504** | **0.0456** | **0.0401** | **0.482** | **12.66** | **21.46** |
| Uncertainty [2] | 0.0017 | 0.012 | 0.0007 | 0.0010 | 0.006 | 0.05 | 0.08 |

| Element | Mo | Cu | Co | V | Nb | W |
|---|---|---|---|---|---|---|
| **Value** [1] | **0.169** | **0.138** | **0.0475** | **0.0188** | **0.140** | **4.17** |
| Uncertainty [2] | 0.003 | 0.003 | 0.0012 | 0.0008 | 0.002 | 0.05 |

## Definitions

[1] 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.

[2] 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:

on 3rd December 2016

MBH ANALYTICAL LIMITED _____

L Maxim

## Method of Preparation

This reference material was produced by continuous casting, and is presented as-cast. The details of the preparation process are unknown.

## Sampling

Milled samples for chemical analysis were taken from several positions within the batch. In addition, at least 10% of all samples were selected for homogeneity checking.

## Homogeneity

Samples representative of the batch were checked for uniformity using an optical emission spectrometer. Multiple measurements were taken from each surface under test.

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

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.

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 linishing, 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.

For optical emission spectroscopy, 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** | 0.0965 | 1.4800 | 0.0442 | 0.0381 | 0.4705 | 12.570 | 21.330 |
| **2** | 0.0965 | 1.4900 | 0.0447 | 0.0382 | 0.4706 | 12.570 | 21.350 |
| **3** | 0.0970 | 1.4920 | 0.0451 | 0.0386 | 0.4708 | 12.582 | 21.390 |
| **4** | 0.0978 | 1.5010 | 0.0451 | 0.0387 | 0.4740 | 12.600 | 21.455 |
| **5** | 0.0990 | 1.5010 | 0.0452 | 0.0398 | 0.4762 | 12.615 | 21.461 |
| **6** | 0.0994 | 1.5030 | 0.0455 | 0.0398 | 0.4780 | 12.630 | 21.464 |
| **7** | 0.0996 | 1.5086 | 0.0456 | 0.0402 | 0.4788 | 12.634 | 21.475 |
| **8** | 0.0999 | 1.5100 | 0.0458 | 0.0408 | 0.4801 | 12.645 | 21.503 |
| **9** | 0.1002 | 1.5110 | 0.0459 | 0.0414 | 0.4821 | 12.680 | 21.510 |
| **10** | 0.1010 | 1.5128 | 0.0459 | 0.0417 | 0.4860 | 12.688 | 21.518 |
| **11** | 0.1013 | 1.5182 | 0.0466 | 0.0418 | 0.4890 | 12.722 | 21.533 |
| **12** | 0.1037 | 1.5250 | 0.0467 | 0.0418 | 0.4910 | 12.734 | 21.570 |
| **13** | 0.1039 | | 0.0470 | | 0.4937 | 12.750 | |
| **14** | | | | | 0.5030 | | |
| **Mean** | **0.0997** | **1.5044** | **0.0456** | **0.0401** | **0.4817** | **12.655** | **21.463** |
| **Std Dev** | 0.0024 | 0.0126 | 0.0008 | 0.0014 | 0.0098 | 0.066 | 0.073 |
| **C (95%)** | 0.0015 | 0.0080 | 0.0005 | 0.0009 | 0.0056 | 0.038 | 0.047 |

| Sample | Mo | Cu | Co | V | Nb | W |
|---|---|---|---|---|---|---|
| **1** | 0.1615 | 0.1306 | 0.0441 | 0.0166 | 0.1341 | 4.100 |
| **2** | 0.1627 | 0.1311 | 0.0449 | 0.0168 | 0.1360 | 4.110 |
| **3** | 0.1653 | 0.1337 | 0.0450 | 0.0175 | 0.1375 | 4.116 |
| **4** | 0.1655 | 0.1350 | 0.0453 | 0.0177 | 0.1379 | 4.117 |
| **5** | 0.1669 | 0.1371 | 0.0462 | 0.0185 | 0.1385 | 4.133 |
| **6** | 0.1680 | 0.1380 | 0.0469 | 0.0188 | 0.1402 | 4.201 |
| **7** | 0.1694 | 0.1384 | 0.0471 | 0.0191 | 0.1406 | 4.205 |
| **8** | 0.1701 | 0.1391 | 0.0474 | 0.0196 | 0.1420 | 4.212 |
| **9** | 0.1710 | 0.1400 | 0.0479 | 0.0197 | 0.1420 | 4.215 |
| **10** | 0.1718 | 0.1402 | 0.0482 | 0.0197 | 0.1422 | 4.221 |
| **11** | 0.1720 | 0.1420 | 0.0495 | 0.0199 | 0.1437 | 4.221 |
| **12** | 0.1724 | 0.1420 | 0.0496 | 0.0199 | 0.1450 | 4.224 |
| **13** | 0.1740 | 0.1430 | 0.0498 | 0.0204 | 0.1460 | |
| **14** | | | 0.0498 | | | |
| **15** | | | 0.0504 | | | |
| **Mean** | **0.1685** | **0.1377** | **0.0475** | **0.0188** | **0.1404** | **4.173** |
| **Std Dev** | 0.0039 | 0.0040 | 0.0021 | 0.0013 | 0.0035 | 0.052 |
| **C (95%)** | 0.0024 | 0.0024 | 0.0011 | 0.0008 | 0.0021 | 0.033 |

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, UK | UKAS accreditation | 0239 |
| Sheffield Analytical Services | Sheffield, UK | UKAS accreditation | 0012 |
| Metals Technology (Testing) Ltd | Sheffield, UK | UKAS accreditation | 0963 |
| Universal Scientific Laboratory Pty Ltd | Milperra, Australia | NATA accreditation | 0492 |
| Genitest Inc. | Montreal, QC, Canada | PRI accreditation | 123077 |
| Shanghai JinYi Test Technology Co. Ltd | Shanghai, China | CNAL accreditation | 0783 |
| Shandong Metallurgical & Science Research | Jinan, Shandong, China | CNAS accreditation | 1461 |
| Bureau Veritas CPS Ltd | Chennai, India | NABL accreditation | 0025 |
| TCR Engineering Services PVT. Ltd. | Mumbai, India | NABL accreditation | 0367 |
| Raghavendra Spectro Metallurgical Laboratory | Bangalore, India | NABL accreditation | 0371 |
| Instytut Metalurgii Zelaza | Gliwice, Poland | PCA accreditation | AB554 |
| TEC Eurolab SRL | Modena, Italy | ACCREDIA accreditation | 52 |
| Mineral and Metallurgical Laboratories | Bangalore, India | | |
| AMG Superalloys UK Ltd | Rotherham, UK | | |
| Analyticka Laborator Lithea sro | Brno, Czech Republic | | |
| Coleshill Laboratories Ltd | Coleshill, UK | | |

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 | - | - | 1-13 | combustion (infra-red detection) |
| Silicon | 3, 6-9 | - | 1, 4, 5, 10, 11 | gravimetric (perchloric acid) |
| | | | 2, 12 | photometric (molybdenum blue) |
| Sulfur | 6 | - | 1-5 7-13 | combustion (IR or volumetric detection) |
| Phosphorus | 1, 2, 7, 9, 10, 12 | - | 3, 4, 6 | photometric (molybdenum blue) |
| | | | 5, 8, 11 | volumetric (alkalimetric) |
| Manganese | 1-5, 7, 10, 12, 13 | 8, 11 | 6, 9 | photometric (periodate) |
| | | | 14 | volumetric (arsenite) |
| Nickel | 1, 2, 4, 10, 12, 13 | 3 | 5, 7, 8, 11, 14 | gravimetric (dimethyl glyoxime) |
| | | | 6 | photometric (dimethyl glyoxime) |
| | | | 9 | volumetric (dimethyl glyoxime, EDTA) |
| Chromium | 4, 10-12 | - | 1-3, 5-9 | volumetric (ferrous ammonium sulfate) |
| Molybdenum | 1, 2, 5, 7, 8, 10, 12 | 4, 6 | 3, 9, 11, 13 | photometric (thiocyanate) |
| Copper | 2, 3, 5, 6, 8, 10, 11 | 1, 7, 13 | 4, 9 | photometric (BCO) |
| | | | 12 | volumetric (thiosulfate) |
| Cobalt | 1, 3, 5, 7-10, 14, 15 | 2, 4, 6 | 11 | photometric (2β-naphthol) |
| | | | 12 | volumetric (iodine) |
| | | | 13 | gravimetric (oxide) |
| Vanadium | 1-3, 6-12 | 5 | 4, 13 | volumetric (ferrous ammonium sulfate) |
| Niobium | 1-8, 10, 11 | 13 | 9 | gravimetric (N-benzoylPH) |
| | | | 12 | photometric (chlorosulfophenol) |
| Tungsten | 2, 4-8, 11 | 1 | 3, 10 | gravimetric (cinchonine) |
| | | | 9, 12 | photometric (thiocyanate) |

## Notes

This Certified Reference Material has been produced and certified in accordance with the requirements of ISO Guide 34-2009, ISO Guide 31-2015 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 ~8mm 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 December 2036, although we reserve the right to make changes as issue revisions, in the intervening period.

This material is also available in the form of chippings

The manufacture, analysis and certification of this product were supervised by L Maxim, Technical Director, MBH Analytical Ltd.

The material to which this certificate of analysis refers is supplied subject to our general conditions of sale.