MAIN EDITION

# BUREAU OF ANALYSED SAMPLES LTD

BRITISH CHEMICAL STANDARD CERTIFIED REFERENCE MATERIAL

# CERTIFICATE OF ANALYSIS

# BCS/SS-CRM No. 401/2

# LOW ALLOY STEEL

**Prepared under rigorous laboratory conditions and, AFTER CERTIFICATION ANALYSIS IN GREAT BRITAIN AND SWEDEN, issued by the Bureau of Analysed Samples Ltd.**

## CO-OPERATING ANALYSTS (ALL ELEMENTS EXCEPT NITROGEN)

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**GOVERNMENT DEPARTMENT**

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11 SHAW, D.H., Howmet Alloys International, Exeter.

## CO-OPERATING ANALYSTS (NITROGEN ONLY)

1 JONES, S.J., Ridsdale & Co Ltd., Middlesbrough.
2 CROCKER F.H. & ATKINSON, M.J. Pattinson & Stead (2005) Ltd., Middlesbrough.
3 LINDKVIST, L., Kanthal AB., Hallstahammar, Sweden
4 SCRIMSHIRE, P.J., IncoTest, Hereford.
5, 7 GUSTAVSSON, I, Gustavsson Consulting, Sweden.
6 COFFEY, R. N Element Teesside, Middlesbrough.

## ANALYSES

Mean of 4 values - mass content in %.

| Lab No. | C | Si | Mn | P | S | Cr | Mo | Ni | Al | Co | Cu | V |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | … | … | … | … | … | 0.132 | 0.503 | 0.021 | 0.074 | 0.0048 | 0.102 | 0.485 |
| 2 | … | … | … | … | … | 0.139 | 0.492 | 0.020 | 0.074 | 0.0043 | 0.104 | 0.494 |
| 3 | 0.945 | 0.596 | 1.199 | 0.0256 | 0.0085 | 0.144 | 0.503 | 0.017 | 0.069 | 0.0036 | 0.101 | 0.498 |
| 4 | 0.920 | 0.602 | 1.212 | 0.0253 | 0.0069 | … | … | … | … | … | 0.104 | 0.500 |
| 5 | 0.934 | 0.617 | 1.200 | 0.0269 | 0.0081 | … | … | … | … | … | … | … |
| 6 | 0.943 | 0.602 | 1.183 | 0.0260 | 0.0072 | … | … | … | … | 0.0037 | … | … |
| 7 | 0.925 | 0.590 | 1.205 | 0.0265 | 0.0083 | … | … | … | … | … | … | … |
| 8 | 0.936 | 0.618 | 1.193 | 0.0294 | 0.0082 | … | … | … | … | … | … | … |
| 9 | … | … | … | … | … | 0.141 | 0.504 | 0.021 | 0.080 | 0.0046 | 0.101 | 0.508 |
| 10 | 0.941 | 0.589 | 1.187 | 0.0259 | 0.0077 | 0.137 | 0.483 | 0.017 | 0.073 | 0.0036 | 0.098 | 0.503 |
| 11 | … | … | … | … | … | 0.135 | 0.483 | 0.019 | 0.076 | 0.0050 | 0.098 | 0.484 |
| **$M_M$** | **0.935** | **0.602** | **1.197** | **0.0265** | **0.0078** | **0.138** | **0.495** | **0.019** | **0.074** | **0.0042** | **0.101** | **0.496** |
| $s_M$ | 0.010 | 0.012 | 0.011 | 0.0014 | 0.0006 | 0.005 | 0.011 | 0.002 | 0.004 | 0.0006 | 0.003 | 0.009 |
| $s_W$ | 0.008 | 0.005 | 0.007 | 0.0008 | 0.0004 | 0.002 | 0.006 | 0.001 | 0.002 | 0.0002 | 0.002 | 0.006 |

| Lab No. | N |
|---|---|
| 1 | 0.0153 |
| 2 | 0.0153 |
| 3 | 0.0161 |
| 4 | 0.0157 |
| 5 | 0.0168 |
| 6 | 0.0160 |
| 7 | 0.0163 |
| | **0.0159** |
| | 0.0006 |
| | 0.0002 |

**$M_M$:** Mean of the laboratory mean values. **$s_M$**: standard deviation of the laboratory mean values. **$s_W$**: average within laboratory standard deviation.

## CERTIFIED VALUES (Cv)

mass content in %

| | C | Si | Mn | P | S | Cr | Mo | Ni | Al | Co | Cu | V | N |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Cv** | **0.935** | **0.602** | **1.197** | **0.0265** | **0.0078** | **0.138** | **0.495** | **0.019** | **0.074** | **0.0042** | **0.101** | **0.496** | **0.0159** |
| C(95%) | 0.009 | 0.011 | 0.010 | 0.0013 | 0.0006 | 0.005 | 0.011 | 0.002 | 0.004 | 0.0006 | 0.003 | 0.009 | 0.0005 |

The half width confidence interval, C(95%), is an expression of the uncertainty of the certified value. C(95%) = $\frac{t \times s_M}{\sqrt{n}}$ where "t" is the appropriate two sided Student's t value at the 95% confidence level for "n" acceptable mean values. For further information regarding the confidence interval for the certified value see ISO Guide 35.

NB: Although widely accepted within the industry "mass content in %" is neither an SI nor an IUPAC supported quantity. Multiplication of the certified value (Cv) by $10^4$ will yield the value in µg/g.



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# BCS/SS-CRM No. 401/2
## LOW ALLOY STEEL
### NOTES ON METHODS USED

**CARBON**

Analyst No. 3 determined carbon by non-aqueous titration according to the British Standard Carbon Method 4*. Nos. 4, 5, 6, 8, and 10 used high frequency combustion/infrared absorption. No. 7 used the gravimetric method BS 6200:3.8.1:1985.

**SILICON**

Analysts Nos. 3, 5, 6, 7 and 10 determined silicon by double dehydration with perchloric acid, according to the British Standard Silicon Method 1*. No. 4 determined silicon photometrically as molybdenum blue, using the Standard Method BS 6200:3.26.3:1987. No.8 used Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES).

**MANGANESE**

Analysts Nos. 3, 4, 5 and 7 determined manganese photometrically after oxidation with periodate according to the British Standard Manganese Method 2*. Nos. 6, 8 and 10 used ICP-OES.

**PHOSPHORUS**

Analysts Nos. 3, 4, 5, 6 and 7 determined phosphorus photometrically as phosphovanadomolybdate. Nos. 3, 5 and 6 used the Standard Method BS 6200:3.24.1:1985. Nos. 8 and 10 used ICP-OES.

**SULPHUR**

Analyst No. 3 determined sulphur gravimetrically according to the British Standard Sulphur Method 1*. Nos. 4, 5, 6, 8 and 10 used high frequency combustion/infrared absorption. No. 7 determined sulphur titrimetrically with borate after combustion of the sample.

**CHROMIUM**

Analyst No. 1 determined chromium using Flame Atomic Absorption Spectrometry (FAAS). Nos. 2, 9, 10 and 11 used ICP-OES. No. 3 determined chromium titrimetrically after oxidation with persulphate/silver nitrate using the Standard Method BS 6200:3.10.1:1985.

**MOLYBDENUM**

Analyst No. 1 determined molybdenum using FAAS. Nos. 2, 9, 10 and 11 used ICP-OES. No. 3 determined molybdenum photometrically as the oxythiocyanate according to the British Standard Molybdenum Method 1*.

**NICKEL**

Analysts Nos. 1, 2 and 3 determined nickel using FAAS. Nos. 9, 10 and 11 used ICP-OES.

**ALUMINIUM**

Analysts Nos. 1 and 3 determined aluminium using FAAS. Nos. 2, 9, 10 and 11 used ICP-OES.

**COBALT**

Analysts Nos. 1, 3 and 6 determined cobalt using FAAS. Nos. 2, 9, 10 and 11 used ICP-OES.

**COPPER**

Analysts Nos. 1, 2, 3 and 4 determined copper using FAAS. Nos. 9, 10, and 11 used ICP-OES.

**VANADIUM**

Analyst No. 1 determined vanadium using FAAS. Nos. 2, 9, 10 and 11 used ICP-OES. Nos. 3 and 4 determined vanadium titrimetrically, No. 3 according to the Analoid Method No. 34

**NITROGEN**

Analysts Nos. 1 and 2 used an acid base titration after steam distillation following a Kjeldahl type digestion. Analysts Nos. 3 and 7 used high frequency combustion/infrared absorption calibrated with pure chemicals whilst Nos. 4, 5 and 6 also used high frequency combustion/infrared absorption but calibrated with a range of carefully selected CRMs (see the Traceability section below for details).

*Methods for Sampling and Analysis of Iron, Steel and other Ferrous Metals, B.S. Handbook No. 19, published 1970 by the British Standards Institute, London.

# BCS/SS-CRM No. 401/2
## LOW ALLOY STEEL

## DESCRIPTION OF SAMPLE

British Chemical Standard BCS-CRM 401/2 is sold in the form of chips passing a nominal 1700µm aperture sieve from which the fines passing a nominal 250µm aperture sieve have been removed. It is supplied in bottles containing 100g.

Spectroscopic Standard SS-CRM 401/2 is sold in the form of 38mm diameter discs.

The preparation of representative samples for chemical analysis and the certification by co-operative analysis was undertaken by Bureau of Analysed Samples Ltd.

Bureau of Analysed Samples Ltd is a United Kingdom Accreditation Service (UKAS) Accredited Reference Material Producer, No 4004, and, as the Producer of BCS/SS-CRM 401/2 as defined in BS EN ISO 17034, is fully responsible for assigning the certified values and their uncertainties in accordance with BS EN ISO 17034 and ISO Guide 35.

## INTENDED USE

BCS-CRM 401/2 is intended for the verification of analytical methods, such as those used by the participating laboratories, for the calibration of analytical instruments, for establishing values for secondary reference materials and for training purposes.

In order to ensure that a fully representative sample is taken users should take not less than the minimum weight stated on the certificate for that element, this being the lowest sample mass used by any of the Co-operating Analysts contributing to the certified value. Users of this material should be aware that the use of a smaller sub-sample size may invalidate the certified values and the associated 95% confidence limits.

The sample should be mixed thoroughly before each use.

SS-CRM 401/2 is intended for establishing and checking the calibration of Optical Emission and X-Ray Spectrometers for the analysis of similar materials. The "as received" working surface of the sample should be linished before use to remove any protective coating. An area 6mm in diameter in the centre of the disc should be avoided for optical emission spectrometry. Users of optical emission spectrometry should also be aware that a minimum of three sparks, made on separate, clean locations across the face of the disc, should be made in order to establish a reliable mean value.

## STABILITY

BCS-CRM 401/2 will remain stable provided that the bottle remains sealed and is stored in a dry atmosphere. Once the bottle has been opened the lid should be secured immediately after use.

SS-CRM 401/2 will remain stable provided that it is not subjected to excessive heat (e.g. during preparation of the working surface).

## TRACEABILITY

The characterisation of this material has been achieved by chemical analysis involving inter-laboratory study, each laboratory using the method of their choice, details of which are given above.

Most of the analytical methods used in the characterisation of this CRM were either international or national standard methods or methods which are technically equivalent. With the exception of C, S and N all laboratories used either stoichiometric analytical techniques or methods which were calibrated predominantly against pure metals or stoichiometric compounds, ensuring traceability of the individual results to the SI.

For C and S, which were certified in 1991, results have been obtained using stoichiometric analytical techniques and high frequency combustion/infrared absorption, methods in which the calibration was not stated but would almost certainly have been obtained using CRMs.

In the case of N, which was certified in 2018, results have been obtained using stoichiometric analytical techniques or by thermal conductimetric methods which were calibrated against either pure stoichiometric compounds or by calibration using five certified reference materials carefully selected to be similar in composition to BCS/SS-CRM 401/2 and all of which demonstrate full unbroken traceability to the SI. Different Analysts used different combinations of CRMs. The CRMs used were as follows: BCS-CRMs 402/2, 404/2, 409/2 and 410/2 and ECRMs 035-2 and 090-1.

## MEASUREMENT UNCERTAINTY

The uncertainty of each of the certified values of BCS/SS-CRM 401/2 has been established by multiplying the standard error arising from the chemical analysis by the appropriate two sided Student's t value at the 95% confidence level for the number of results. Homogeneity has been assessed and found to be acceptable. Homogeneity has not, therefore, been included in the calculated measurement uncertainty. The stability of this CRM and its transportation also make negligible contributions to the overall uncertainty of the certified values.

## COMMUTABILTY

BCS-CRM 401/2 is intended to be used in the same physical form as that used by the participating analysts and therefore commutability is not of relevance in respect of this CRM.

In the case of SS-CRM 401/2 it has been established that, when using optical emission spectrometers, materials of similar composition from different sources may respond differently. The user should be aware that the metallurgical history of this SS-CRM may not accurately reflect the metallurgical history of the user's own materials.

Further information and advice on this or other Certified Reference Materials or Reference Materials produced by Bureau of Analysed Samples Ltd may be obtained from the address below:

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For BUREAU OF ANALYSED SAMPLES LTD

R P MEERES,
Managing Director
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*Preliminary Edition* …. …. …. …. …. March 1991
*Main Edition* …. …. …. …. …. April 1992
*Revised with C(95%) and $s_W$ values for each certified element* …. …. …. December 2005
*Revised with certified value for nitrogen*. …. …. …. February 2019