

BRAMMER STANDARD COMPANY, INC.

Certificate of Analysis

BS HiCal-1

Certified Reference Material for Calcium Treated Low Alloy Steel

| | Certified Value ¹ | Estimate of Uncertainty ² | Certified Values³ | Certified Value ¹ | Estimate of Uncertainty ² |
|-----------|------------------------------|--------------------------------------|-------------------------------------|------------------------------|--------------------------------------|
| Al | 0.070 | 0.002 | | | |
| As | 0.0022 | 0.0006 | | | |
| C | 0.271 | 0.004 | | | |
| Ca | 0.0140 | 0.0007 | | | |
| Co | 0.0024 | 0.0007 | | | |
| Cr | 1.55 | 0.03 | | | |
| Cu | 0.152 | 0.002 | | | |
| Fe | 91.9 | 0.4 | | | |
| Mn | 1.00 | 0.02 | | | |
| Mo | 0.379 | 0.009 | | | |
| Ni | 3.28 | 0.02 | | | |
| S | 0.0007 | 0.0003 | | | |
| Si | 1.29 | 0.02 | | | |
| Ti | 0.0037 | 0.0003 | | | |
| V | 0.0027 | 0.0006 | | | |

Informational Values^{3,4}

| | | | | |
|-------------|-------------|-------------|-----------|-------------|
| B (0.0001) | Mg (0.0003) | Nb (0.002) | P (0.007) | Pb (0.0005) |
| Sn (0.0002) | W (0.0009) | Zr (0.0008) | | |

¹ For each element, the certified value listed is the present best estimate of the true value based on the mean of the weighted results of an interlaboratory testing program. See page 3 for more information on its calculation.

² For each element, the uncertainty listed is based on a statistical evaluation of the contributions of homogeneity and the interlaboratory testing program. See page 3 for more information on its calculation.

³ Values are given in weight percent. Values in brackets are reported by difference.

⁴ Values in parentheses are not certified and are provided for information only.

Trace element information values for N and O are shown on page 3.

The requirements of ISO Guides 31, 34, and 35 were followed for the preparation of this Certified Reference Material and certificate of analysis. This is a Certified Reference Material as defined by ISO Guide 30.

BS HiCal-1 * Code for method Certified values listed as weight percent

| Analysis | * | Al | * | As | * | C | * | Ca | * | Co | * | Cr | * | Cu | * | Fe |
|--------------------|----|--------------|----|---------------|---|--------------|----|---------------|----|---------------|----|-------------|----|--------------|----|-------------|
| 1 | 4 | 0.061433 | 15 | 0.00130 | 1 | 0.261 | 4 | 0.012667 | 5 | 0.001467 | 3 | 1.51 | 5 | 0.140 | 13 | 91.56867 |
| 2 | 5 | 0.068433 | 4 | 0.0016 | 1 | 0.265487 | 4 | 0.012933 | 4 | 0.00158 | 14 | 1.52 | 3 | 0.148333 | 4 | 91.80333 |
| 3 | 3 | 0.0706 | 5 | 0.001733 | 1 | 0.266 | 4 | 0.012933 | 5 | 0.0017 | 3 | 1.52 | 3 | 0.15 | 16 | [91.80667] |
| 4 | 4 | 0.070967 | 5 | 0.0019 | 1 | 0.267667 | 4 | 0.013267 | 4 | 0.0018 | 3 | 1.52 | 4 | 0.150 | 10 | 91.84667 |
| 5 | 4 | 0.071033 | 3 | 0.0046 | 3 | 0.268 | 4 | 0.013333 | 5 | 0.00182 | 4 | 1.520333 | 14 | 0.151667 | 16 | [91.91013] |
| 6 | 4 | 0.071033 | 4 | 0.0047 | 3 | 0.269 | 3 | 0.01375 | 4 | 0.0027 | 3 | 1.53 | 4 | 0.1517 | 16 | [91.94] |
| 7 | 14 | 0.072367 | 9 | 0.0048 | 1 | 0.271333 | 3 | 0.0138 | 4 | 0.003167 | 4 | 1.5471 | 10 | 0.152 | 4 | 91.95667 |
| 8 | 3 | 0.073 | | | 3 | 0.273 | 14 | 0.014233 | 3 | 0.0035 | 4 | 1.55 | 8 | 0.152333 | 14 | 92.0 |
| 9 | 4 | 0.073633 | | | 1 | 0.273667 | 3 | 0.01472 | 14 | 0.0037 | 10 | 1.55 | 4 | 0.153333 | 16 | [92.15] |
| 10 | 3 | 0.074 | | | 1 | 0.275333 | 4 | 0.01501 | 3 | 0.004133 | 4 | 1.576333 | 4 | 0.154333 | 3 | 92.23 |
| 11 | 10 | 0.0742 | | | 1 | 0.275333 | 4 | 0.0152 | | | 13 | 1.577333 | 7 | 0.155 | | |
| 12 | 3 | 0.083267 | | | 1 | 0.276433 | | | | | 4 | 1.5774 | 4 | 0.155067 | | |
| 13 | | | | | | | | | | | 10 | 1.58 | 3 | 0.156 | | |
| 14 | | | | | | | | | | | 4 | 1.605333 | 3 | 0.158 | | |
| 15 | | | | | | | | | | | | | 10 | 0.158 | | |
| Average | | 0.0705 | | 0.00222 | | 0.2713 | | 0.01404 | | 0.00239 | | 1.5515 | | 0.1524 | | 91.948 |
| Std dev | | 0.0022 | | 0.00019 | | 0.0041 | | 0.00060 | | 0.00016 | | 0.0079 | | 0.0035 | | 0.039 |
| H | | 0.0018 | | 0.00040 | | 0.0041 | | 0.0008 | | 0.00041 | | 0.014 | | 0.0029 | | 0.51 |
| U ₁ | | 0.0028 | | 0.00044 | | 0.0058 | | 0.0010 | | 0.00044 | | 0.016 | | 0.0045 | | 0.51 |
| t-statistic | | 2.20 | | 2.45 | | 2.20 | | 2.23 | | 2.26 | | 2.16 | | 2.14 | | 2.26 |
| U ₂ | | 0.0062 | | 0.0011 | | 0.013 | | 0.0023 | | 0.0010 | | 0.034 | | 0.010 | | 1.15 |
| U ₃ | | 0.0018 | | 0.00041 | | 0.0037 | | 0.00068 | | 0.00031 | | 0.0090 | | 0.0025 | | 0.36 |
| Certified | | 0.070 | | 0.0022 | | 0.271 | | 0.0140 | | 0.0024 | | 1.55 | | 0.152 | | 91.9 |
| Uncertainty | | 0.002 | | 0.0006 | | 0.004 | | 0.0007 | | 0.0007 | | 0.03 | | 0.002 | | 0.4 |
| Tolerance | | 0.006 | | 0.0014 | | 0.013 | | 0.0023 | | 0.0015 | | 0.06 | | 0.010 | | 1.2 |

| Analysis | * | Mn | * | Mo | * | Ni | * | S | * | Si | * | Ti | * | V |
|--------------------|----|-------------|----|--------------|----|-------------|---|---------------|----|-------------|----|---------------|----|---------------|
| 1 | 3 | 0.975 | 3 | 0.351333 | 3 | 3.206667 | 1 | 0.000233 | 3 | 1.25 | 5 | 0.003047 | 5 | 0.002 |
| 2 | 3 | 0.981 | 5 | 0.352 | 3 | 3.21 | 1 | 0.0003 | 14 | 1.25333 | 5 | 0.003133 | 4 | 0.0022 |
| 3 | 3 | 0.983 | 3 | 0.361 | 4 | 3.2445 | 1 | 0.0003 | 4 | 1.269 | 5 | 0.003433 | 5 | 0.002267 |
| 4 | 14 | 0.983 | 3 | 0.365 | 4 | 3.254667 | 1 | 0.000433 | 3 | 1.27 | 3 | 0.003533 | 5 | 0.00245 |
| 5 | 4 | 0.987 | 4 | 0.370 | 13 | 3.265 | 1 | 0.000433 | 10 | 1.30667 | 3 | 0.0036 | 4 | 0.0027 |
| 6 | 3 | 1.000 | 3 | 0.370 | 14 | 3.283333 | 1 | 0.000467 | 4 | 1.31333 | 4 | 0.003633 | 10 | 0.0032 |
| 7 | 8 | 1.000333 | 4 | 0.371933 | 4 | 3.285333 | 1 | 0.000667 | 6 | 1.31833 | 4 | 0.003733 | 3 | 0.0038 |
| 8 | 7 | 1.005 | 4 | 0.372667 | 4 | 3.292667 | 1 | 0.000937 | 3 | 1.32 | 4 | 0.0038 | 14 | 0.003867 |
| 9 | 4 | 1.015667 | 10 | 0.375333 | 10 | 3.296667 | 3 | 0.0051 | 4 | 1.32233 | 14 | 0.004067 | 10 | 0.004733 |
| 10 | 4 | 1.0179 | 10 | 0.379 | 4 | 3.300 | 3 | 0.0053 | 4 | 1.33937 | 3 | 0.0041 | | |
| 11 | 10 | 1.020 | 4 | 0.3897 | 10 | 3.32 | | | 4 | 1.34333 | 3 | 0.0042 | | |
| 12 | 4 | 1.026667 | 7 | 0.396 | 3 | 3.34 | | | 10 | 1.370 | 10 | 0.0042 | | |
| 13 | 4 | 1.029333 | 4 | 0.400667 | 3 | 3.37 | | | 6 | 1.39 | 10 | 0.0047 | | |
| 14 | 4 | 1.035667 | 14 | 0.401 | 4 | 3.433333 | | | | | | | | |
| 15 | 10 | 1.06 | | | | | | | | | | | | |
| Average | | 1.0034 | | 0.3795 | | 3.282 | | 0.000724 | | 1.2923 | | 0.00374 | | 0.00273 |
| Std dev | | 0.0068 | | 0.0046 | | 0.021 | | 0.000081 | | 0.0070 | | 0.00026 | | 0.00022 |
| H | | 0.010 | | 0.0051 | | 0.025 | | 0.00029 | | 0.01204 | | 0.00048 | | 0.00043 |
| U ₁ | | 0.012 | | 0.0069 | | 0.032 | | 0.00030 | | 0.014 | | 0.00054 | | 0.00048 |
| t-statistic | | 2.14 | | 2.16 | | 2.16 | | 2.26 | | 2.18 | | 2.18 | | 2.31 |
| U ₂ | | 0.026 | | 0.015 | | 0.070 | | 0.00067 | | 0.030 | | 0.0012 | | 0.0011 |
| U ₃ | | 0.0070 | | 0.0040 | | 0.019 | | 0.00021 | | 0.0084 | | 0.00033 | | 0.00037 |
| Certified | | 1.00 | | 0.379 | | 3.28 | | 0.0007 | | 1.29 | | 0.0037 | | 0.0027 |
| Uncertainty | | 0.02 | | 0.009 | | 0.02 | | 0.0003 | | 0.02 | | 0.0003 | | 0.0006 |
| Tolerance | | 0.04 | | 0.022 | | 0.07 | | 0.0007 | | 0.05 | | 0.0012 | | 0.0013 |

BS HiCal-1 * Code for method Informational values listed as weight percent

| Analysis | * | B | * | Mg | * | Nb | * | P | * | Pb | * | Sn | * | W | * | Zr |
|-----------------|---|-----------------|---|-----------------|----|----------------|----|----------------|---|-----------------|---|-----------------|----|-----------------|----|-----------------|
| 1 | 3 | 0.00005 | 4 | 0.0003 | 5 | 0.0003 | 5 | 0.00317 | 5 | 0.0003 | 4 | 0.0001 | 5 | 0.000107 | 5 | 0.0001 |
| 2 | 4 | 0.000133 | 5 | 0.00043 | 5 | 0.00038 | 7 | 0.00364 | 9 | 0.000327 | 3 | 0.0001 | 5 | 0.0008 | 5 | 0.00023 |
| 3 | 7 | 0.000213 | | | 5 | 0.00042 | 4 | 0.004383 | 4 | 0.0005 | 4 | 0.0001 | 4 | 0.000833 | 5 | 0.00034 |
| 4 | | | | | 4 | 0.0014 | 4 | 0.0046 | 4 | 0.000607 | 5 | 0.000133 | 5 | 0.000893 | 3 | 0.001 |
| 5 | | | | | 3 | 0.0017 | 4 | 0.0053 | 3 | 0.0007 | 5 | 0.00014 | 4 | 0.002933 | 5 | 0.001133 |
| 6 | | | | | 14 | 0.0026 | 3 | 0.006433 | 3 | 0.0007 | 5 | 0.000157 | 3 | 0.0044 | 4 | 0.001167 |
| 7 | | | | | 4 | 0.002633 | 10 | 0.0072 | | | 9 | 0.0009 | 14 | 0.004933 | 10 | 0.0012 |
| 8 | | | | | 10 | 0.0028 | 10 | 0.0075 | | | | | 3 | 0.0072 | 3 | 0.0013 |
| 9 | | | | | 3 | 0.0034 | 4 | 0.0079 | | | | | | | | |
| 10 | | | | | | | 3 | 0.0087 | | | | | | | | |
| 11 | | | | | | | 4 | 0.0093 | | | | | | | | |
| 12 | | | | | | | 14 | 0.009767 | | | | | | | | |
| 13 | | | | | | | 3 | 0.0107 | | | | | | | | |
| Average | | 0.000132 | | 0.000300 | | 0.0017 | | 0.007 | | 0.00052 | | 0.00023 | | 0.00088 | | 0.0008 |
| Std dev | | 0.000063 | | 0.000027 | | 0.0064 | | 0.039 | | 0.00088 | | 0.00015 | | 0.00010 | | 0.0018 |
| H | | 0.00020 | | 0.00023 | | 0.00036 | | 0.00061 | | 0.00026 | | 0.00022 | | 0.00030 | | 0.0003 |
| U ₁ | | 0.00021 | | 0.00023 | | 0.0064 | | 0.039 | | 0.00091 | | 0.00027 | | 0.00032 | | 0.0018 |
| t-statistic | | 4.30 | | 12.71 | | 2.31 | | 2.18 | | 2.57 | | 2.45 | | 2.36 | | 2.36 |
| U ₂ | | 0.00091 | | 0.0030 | | 0.015 | | 0.085 | | 0.0023 | | 0.00065 | | 0.00075 | | 0.0042 |
| U ₃ | | 0.00052 | | 0.0021 | | 0.0049 | | 0.024 | | 0.00096 | | 0.00025 | | 0.00027 | | 0.0015 |
| (Informational) | | (0.0001) | | (0.0003) | | (0.002) | | (0.007) | | (0.0005) | | (0.0002) | | (0.0009) | | (0.0008) |

For each element, in accordance with the requirements of ISO Guides 34 and 35, an effort must be made to account for the effects on the certified value of the uncertainty estimate from homogeneity testing (H) and the uncertainties of the contributing laboratories. The average (A) is calculated using a weighted mean where the reciprocal of the square of each laboratory's combined uncertainty (C_L), calculated from its standard deviation (S_L) and its uncertainty estimate (U_L), is used as the weight (W_L) for its mean (M_L). The standard deviation (S) is calculated as the square root of the reciprocal of the sum of the weights. U₁ is the combined uncertainty from homogeneity and labs. U₂ is U₁ multiplied by the coverage factor (95 % t-statistic). U₃ is U₂ divided by the square root of the number of determinations (n). Thus:

$$C_L = \sqrt{S_L^2 + U_L^2} \quad W_L = \frac{1}{C_L^2} \quad A = \frac{\sum_{i=1}^n W_L M_L}{\sum_{i=1}^n W_L} \quad S = \frac{1}{\sqrt{\sum_{i=1}^n W_L}} \quad U_1 = \sqrt{H^2 + S^2} \quad U_2 = t \times U_1 \quad U_3 = \frac{U_2}{\sqrt{n}}$$

All but the final reported values are taken to two significant figures as determined by each quantity's uncertainty estimate. The final reported Uncertainty is U₃ rounded to one significant figure and represents the half width of the 95 % confidence interval for the **Certified** value. The final reported **Certified** value is A rounded to the same decimal place as the Uncertainty. The Tolerance is the half width of the 95 % confidence interval for measurements rounded to the same decimal place as the Uncertainty. The Uncertainty is a measure of the quality of the **Certified** value. The Tolerance is a measure of the expected performance of an analysis.

For further information regarding the confidence interval for the certified value see ISO Guide 35:2006 section 6.

BS HiCal-1 * Code for analytical method Trace analysis listed as mg/kg (ppm)

| Analysis | * | N | * | O |
|----------|---|------|---|------|
| 1 | 2 | 45.4 | 2 | 2.55 |
| 2 | 2 | 47.7 | 2 | 3.86 |
| 3 | 2 | 49.8 | 2 | 4.05 |

Analytical Method Codes:

| | | |
|---------------------------|---------------------------|---------------------------|
| 1 Combustion (ASTM E1019) | 7 Photometric | 13 Titrimetric |
| 2 Fusion (ASTM E1019) | 8 Flame Atomic Absorption | 14 DCP Atomic Emission |
| 3 Spark Atomic Emission | 9 GF Atomic Absorption | 15 HG Atomic Fluorescence |
| 4 ICP Atomic Emission | 10 X-Ray Fluorescence | 16 Difference |
| 5 ICP Mass Spectrometry | 11 GD Atomic Emission | |
| 6 Gravimetric | 12 GD Mass Spectrometry | |

ICP = Inductively Coupled Plasma GF = Graphite Furnace GD = Glow Discharge
DCP = Direct Current Plasma HG = Hydride Generation

| <u>Laboratory</u> | <u>Location</u> | <u>Registrar</u> | <u>Accreditation</u> |
|---|----------------------|------------------|----------------------|
| Brammer Standard Company, Inc. | Houston, TX | A2LA | 17025, Guide 34 |
| LECO Corporation | St. Joseph, MI | A2LA | 17025 |
| Dirats Laboratories | Westfield, MA | ACLASS | 17025 |
| NSL Analytical | Cleveland, OH | ACLASS | 17025 |
| Luvak Inc. | Boylston, MA | PRI/Nadcap | 17025 |
| National Analysis Center For Iron And Steel | Beijing, China | CNAS | 17025 |
| Laboratory Testing, Inc. | Hatfield, PA | PRI/Nadcap | 17025 |
| Exova | Glendale Heights, IL | A2LA | 17025 |
| Instytut Metalurgii Zelaza | Gliwice, Poland | PCA | AB 554 |

A2LA = American Association for Laboratory Accreditation

ACLASS = ANSI-ASQ National Accreditation Board

BSI = British Standards Institution

CNAS = China National Accreditation Service

NABL = National Accreditation Board for Testing and Calibration Laboratories

Nadcap = National Aerospace and Defense Contractors Accreditation Program

PCA = Polish Center For Accreditation

PRI = Performance Review Institute

SRI = Steel Related Industries

TUV = Technischer Überwachungs-Verein (Technical Inspection Association)

Analysis: Chemical analyses were made on solid pieces from representative samples for the certified portion of the lot in accordance with ASTM Standard Practice E1806. The laboratories participating in the testing followed the requirements of ISO Standard 17025.

Traceability: The following Certified Reference Materials were used to validate the analytical data: 12X352B, 12X32550, 12X44220, 13X31254, 13X32101; 501-320, 501-501, 501-503, 501-504, 501-550, 501-644, 501-673, 501-676, 501-993, 502-414, 502-873; AR 148, 642, 871, 872, 875, 892, 1653; BAS 409, 40-1, 434, 435, 464/1; BS CA-2, CA-5A, 1C, 2C, 11A, 14A, 30D, 56H, 300, 1026, 1030; ECRM 184-1; IARM 28G, 35H, 156B, 210C; IMZ 55/1, 58/1, 130, 139; JK Nr8F, 37; SRM 16F, 33C, 33D, 33E, 55D, 72F, 72G, 107B, 160V, 291, 293, 361, 362, 363, 364, 393, 691, 1228, 1261, 1765, 1766, 3109A, 3163, 3169.

Homogeneity: This Certified Reference Material (CRM) was tested for homogeneity using ASTM Standard Method E826 and found acceptable. It was also examined by spark atomic emission spectrometry and found to be compatible with the following Reference Materials — 501-676; BS CA-2, CA-5, CA-5A, 1C, 2C, 11A, 14A; ECRM 184-1; SRM 72G, 291.

Validity statement: ISO Guide 31 states that the certification should contain an expiration date for all materials where instability has been demonstrated or is considered possible, after which the certified value is no longer guaranteed by the certifying body. The certification of BS HiCal-1 is valid indefinitely. The certification is nullified if this CRM is damaged, contaminated, or otherwise modified.

Source: The cast stock for this CRM was produced by Jey Swen Enterprises; Koahsiung, China.

Form: This CRM is machined in the form of a disc, approximately 38 mm in diameter and 30 mm thick by Brammer Standard Company, Inc.

Use: This CRM is intended for use in spark atomic emission, glow discharge, and x-ray spectrometric methods of analysis. Refer to

ISO Guide 33 for information about the use of Certified Reference Materials.

Certified Area: The certified area of each disc is the portion extending several mm inward from each surface.

Sample Preparation: For best analytical results, use the same method for preparing the analytical surface on all reference materials as used for production specimens. Avoid overheating the sample during surface preparation.

Certificate Number: The unique identification number for this certificate of analysis is HiCal-1-020316. You may obtain information on revisions of certificates from the internet at www.brammerstandard.com.

Safety Notice: A Safety Data Sheet (SDS) is not required for this material. This material will not release or otherwise result in exposure to a hazardous chemical, under normal conditions of use. Inquiries concerning this Reference Material should be directed to:

Brammer Standard Co., Inc. **Phone: (281) 440-9396** **Web: www.brammerstandard.com**
14603 Benfer Road
Houston, Texas 77069-2895 USA **Fax: (281) 440-4432** **Email: contact@brammerstandard.com**

Brammer Standard Company, Inc., is accredited by the American Association For Laboratory Accreditation (A2LA) to ISO Guide 34 as a Reference Material Producer for the production of Certified Reference Materials and Reference Materials (Certificate Number 656.02)

Brammer Standard Company's Chemical Laboratory is accredited by A2LA to ISO Standard 17025. (Certificate Number 656.01)

By Certificate Number 10539, the Quality System of Brammer Standard Company, Inc., is registered to ISO 9001:2008 by National Quality Assurance (NQA), U.S.A.

The scopes of accreditation are listed on the website: www.brammerstandard.com

References:

Brammer Standard Company, Inc., 14603 Benfer Road, Houston, TX 77069-2895
Telephone: (281) 440-9396 Fax: (281) 440-4432 Website: www.brammerstandard.com
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Versions used were those available at the time of testing and characterization

- E826 Standard Practice for Testing Homogeneity of a Metal Lot or Batch in Solid Form by Spark Atomic Emission Spectrometry
- E1019 Standard Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques
- E1806 Standard Practice for Sampling Steel and Iron for Determination of Chemical Composition

ISO Standard 17025:2005 General requirements for the competence of testing and calibration laboratories

ISO Standard 9001:2008 Quality Management Systems - Requirements

ISO Guide 30:1992 Terms and definitions used in connection with reference materials + 2008 amendment

ISO Guide 31:2000 Reference materials - Contents of certificates and labels

ISO Guide 33:2000 Uses of certified reference materials

ISO Guide 34:2009 General requirements for the competence of reference material producers

ISO Guide 35:2006 Reference Materials - General and statistical principles for certification

ASTM documents available from ASTM, 100 Barr Harbor Dr, West Conshohocken, PA, 19428.

ISO Guides and Standards available from Global Engineering - www.global.ihs.com

Other useful documents available from NIST, U.S. Department of Commerce, Gaithersburg, MD 20899.

NIST Special Publication 260-100, Handbook for SRM Users

NIST Special Publication 829, Use of NIST Standard Reference Materials for Decisions on Performance of Analytical Chemical Methods and Laboratories

Certified by: _____ on February 3, 2016.

Beau R. Brammer