

# BRAMMER STANDARD COMPANY, INC.

## Certificate of Analysis

### BS 101/4

Certified Reference Material for Basic Oxygen Furnace Slags<sup>1</sup>

|                                | Certified Value <sup>2</sup> | Estimate of Uncertainty <sup>3</sup> | Certified Values <sup>4</sup> |
|--------------------------------|------------------------------|--------------------------------------|-------------------------------|
| Al <sub>2</sub> O <sub>3</sub> | 0.86                         | 0.04                                 |                               |
| CaO                            | 52.5                         | 0.7                                  |                               |
| K <sub>2</sub> O               | 0.007                        | 0.001                                |                               |
| MgO                            | 4.8                          | 0.2                                  |                               |
| MnO                            | 4.79                         | 0.05                                 |                               |
| Na <sub>2</sub> O              | 0.018                        | 0.004                                |                               |
| P <sub>2</sub> O <sub>5</sub>  | 0.81                         | 0.04                                 |                               |
| S                              | 0.15                         | 0.01                                 |                               |
| SiO <sub>2</sub>               | 16.7                         | 0.3                                  |                               |
| TiO <sub>2</sub>               | 1.16                         | 0.06                                 |                               |

### Informational Values<sup>4,5</sup>

T Fe (13.3)

**Revision:** This certified reference material was originally certified as a reference material in September 1976. It was revised on April 20, 1989 to add Sodium and potassium oxide analysis. The uncertainties were added as a revision on February 02, 2009. This 2013 revision demonstrates stability with additional laboratory testing from ISO 17025 accredited laboratories. Revised values for all elements except S are presented. Na<sub>2</sub>O and K<sub>2</sub>O are now certified values. T. Fe is now informational. The new revised values all fall within the previous uncertainties, proving stability.

<sup>1</sup> This certificate is a revision. For more information on the nature and extent of the revision, see the revision statement above.

<sup>2</sup> 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.

<sup>3</sup> 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.

<sup>4</sup> Values are given in weight percent.

<sup>5</sup> Values in parentheses are not certified and are provided for information only.

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 101/4** \* Code for method Certified values listed as weight percent

| Analysis           | *  | Al <sub>2</sub> O <sub>3</sub> | *  | CaO         | *  | K <sub>2</sub> O | *  | MgO        | *  | MnO         | *  | Na <sub>2</sub> O | *  | P <sub>2</sub> O <sub>5</sub> | * | S           |
|--------------------|----|--------------------------------|----|-------------|----|------------------|----|------------|----|-------------|----|-------------------|----|-------------------------------|---|-------------|
| 1                  | 16 | 0.80                           | 16 | 52.2        | 16 | 0.0060           | 16 | 4.70       | 16 | 4.70        | 16 | 0.0160            | 7  | 0.77                          | 1 | 0.140       |
| 2                  | 16 | 0.85                           | 16 | 52.5        | 16 | 0.0060           | 4  | 4.79       | 16 | 4.70        | 4  | 0.0160            | 7  | 0.78                          | 1 | 0.140       |
| 3                  | 7  | 0.86                           | 16 | 52.6        | 8  | 0.0074           | 16 | 4.80       | 7  | 4.75        | 16 | 0.0190            | 16 | 0.83                          | 1 | 0.150       |
| 4                  | 16 | 0.90                           | 16 | 52.8        | 4  | 0.0076           | 16 | 4.80       | 16 | 4.82        | 8  | 0.0218            | 16 | 0.86                          | 1 | 0.153       |
| 5                  | 16 | 0.90                           | 16 | 52.9        | 16 | 0.0080           | 8  | 4.90       | 16 | 4.86        |    |                   |    |                               | 1 | 0.160       |
| 6                  |    |                                | 16 | 53.0        |    |                  |    |            | 8  | 4.89        |    |                   |    |                               | 1 | 0.160       |
| 7                  |    |                                |    |             |    |                  |    |            |    |             |    |                   |    |                               | 1 | 0.160       |
| Average            |    | 0.863                          |    | 52.66       |    | 0.00705          |    | 4.800      |    | 4.789       |    | 0.01830           |    | 0.809                         |   | 0.1519      |
| Std dev            |    | 0.010                          |    | 0.14        |    | 0.00048          |    | 0.031      |    | 0.028       |    | 0.00096           |    | 0.011                         |   | 0.0029      |
| H                  |    | 0.008                          |    | 0.31        |    | 0.00046          |    | 0.032      |    | 0.032       |    | 0.0007            |    | 0.008                         |   | 0.0024      |
| U <sub>1</sub>     |    | 0.013                          |    | 0.34        |    | 0.00067          |    | 0.045      |    | 0.043       |    | 0.0012            |    | 0.013                         |   | 0.0038      |
| t-statistic        |    | 2.78                           |    | 2.57        |    | 2.78             |    | 2.78       |    | 2.57        |    | 3.18              |    | 3.18                          |   | 2.45        |
| U <sub>2</sub>     |    | 0.036                          |    | 0.88        |    | 0.0019           |    | 0.12       |    | 0.11        |    | 0.0038            |    | 0.042                         |   | 0.0092      |
| U <sub>3</sub>     |    | 0.016                          |    | 0.36        |    | 0.00083          |    | 0.055      |    | 0.045       |    | 0.0019            |    | 0.021                         |   | 0.0035      |
| <b>Certified</b>   |    | <b>0.86</b>                    |    | <b>52.5</b> |    | <b>0.007</b>     |    | <b>4.8</b> |    | <b>4.79</b> |    | <b>0.018</b>      |    | <b>0.81</b>                   |   | <b>0.15</b> |
| <b>Uncertainty</b> |    | <b>0.04</b>                    |    | <b>0.7</b>  |    | <b>0.001</b>     |    | <b>0.2</b> |    | <b>0.05</b> |    | <b>0.004</b>      |    | <b>0.04</b>                   |   | <b>0.01</b> |
| Tolerance          |    | 0.04                           |    | 0.9         |    | 0.002            |    | 0.1        |    | 0.11        |    | 0.004             |    | 0.04                          |   | 0.01        |

| Analysis           | *  | SiO <sub>2</sub> | *  | TiO <sub>2</sub> |
|--------------------|----|------------------|----|------------------|
| 1                  | 16 | 16.39            | 16 | 1.14             |
| 2                  | 16 | 16.50            | 16 | 1.14             |
| 3                  | 6  | 16.50            | 8  | 1.16             |
| 4                  | 16 | 16.60            | 7  | 1.18             |
| 5                  | 16 | 16.80            |    |                  |
| 6                  | 16 | 16.83            |    |                  |
| 7                  | 16 | 16.85            |    |                  |
| 8                  | 16 | 17.00            |    |                  |
| Average            |    | 16.685           |    | 1.156            |
| Std dev            |    | 0.055            |    | 0.013            |
| H                  |    | 0.10             |    | 0.010            |
| U <sub>1</sub>     |    | 0.11             |    | 0.017            |
| t-statistic        |    | 2.36             |    | 3.18             |
| U <sub>2</sub>     |    | 0.27             |    | 0.053            |
| U <sub>3</sub>     |    | 0.10             |    | 0.027            |
| <b>Certified</b>   |    | <b>16.7</b>      |    | <b>1.16</b>      |
| <b>Uncertainty</b> |    | <b>0.3</b>       |    | <b>0.06</b>      |
| Tolerance          |    | 0.3              |    | 0.05             |

**BS 101/4** \* Code for method Informational values listed as weight percent

| Analysis           | *  | T Fe          |
|--------------------|----|---------------|
| 1                  | 16 | 13.26         |
| 2                  | 16 | 13.32         |
| 3                  | 16 | 13.40         |
| Average            |    | 13.330        |
| Std dev            |    | 0.076         |
| H                  |    | 0.08          |
| U <sub>1</sub>     |    | 0.11          |
| t-statistic        |    | 4.30          |
| U <sub>2</sub>     |    | 0.48          |
| U <sub>3</sub>     |    | 0.28          |
| <b>(Certified)</b> |    | <b>(13.3)</b> |

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<sub>L</sub>), calculated from its standard deviation (S<sub>L</sub>) and its uncertainty estimate (U<sub>L</sub>), is used as the weight (W<sub>L</sub>) for its mean (M<sub>L</sub>). The standard deviation (S) is calculated as the square root of the reciprocal of the sum of the weights. U<sub>1</sub> is the combined uncertainty from homogeneity and labs. U<sub>2</sub> is U<sub>1</sub> times the coverage factor (95 % t-statistic). U<sub>3</sub> is U<sub>2</sub> 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 U<sub>3</sub> 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.

**Analytical Method Codes:**

- |                           |                           |                           |
|---------------------------|---------------------------|---------------------------|
| 1 Combustion (ASTM E1019) | 7 Photometric             | 13 Titrimetric            |
| 2 Fusion (ASTM E 1019)    | 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 Wet Method             |
| 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     |                  |                      |
| LECO Corporation                          | St. Joseph, MI  |                  |                      |
| Carpenter Technology Corporation          | Reading, PA     |                  |                      |
| Crobaugh                                  | Cleveland, OH   |                  |                      |
| Andrew S. McCreath & Son, Inc.            | Harrisburg, PA  |                  |                      |
| Coors                                     | Golden, CO      |                  |                      |
| Ledoux                                    | Teaneck, NJ     |                  |                      |
| J&L                                       | Canonsburg, PA  |                  |                      |
| Benxi Iron & Steel Co.,Ltd.               | Liaoning, China | CNAS             | 17025                |
| Shandong Metallurgical Research Insitiute | Jinan, China    | CNAS             | 17025                |

CNAS = China National Accreditation Service

**Analysis:** Chemical analyses were made from representative samples for the certified portion of the lot in accordance with ASTM Standard Practice E 1806. The laboratories participating in the testing generally followed the requirements of ISO Standard 17025 before its existence, although many are now ISO 17025 accredited. Methods of analysis used were those listed on page 2.

**Traceability:** Certified Reference Materials were used to validate the analytical data listed on page 2 — including SRM, ECRM, and BAS samples.

**Homogeneity:** This Certified Reference Material (CRM) was tested for homogeneity using ASTM Standard Method E 826 and found acceptable.

**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 101/4 is valid for at least 20 years at which time stability is verified and a new certificate will be issued. The material needs to be stored in cool, dry conditions to avoid any moisture contamination. The certification is nullified if this CRM is damaged, contaminated, or otherwise modified.

**Source:** The stock for this CRM was produced by Jones and Laughlin Steel, Aliquippa, PA

**Form:** This CRM is bottled with approximately 100 g. of powder by Brammer Standard Company, Inc.

**Use:** This CRM is intended for any method of analysis capable of analyzing powders or pressed powders. Refer to ISO Guide 33 for information about the use of Certified Reference Materials.

**Sample Preparation:** For best results, dry the material for one hour at 105°C and mix well before using. The material has been pulverized to pass a #100 sieve.

**Certificate Number:** The unique identification number for this certificate of analysis is REV101/4-050713. You may obtain information on revisions of certificates from the internet at [www.brammerstandard.com](http://www.brammerstandard.com).

**Safety Notice:** A Material Safety Data Sheet (MSDS) 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:

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14603 Benfer Road  
Houston, Texas 77069-2895 USA

**Phone: (281) 440-9396**  
**Fax: (281) 440-4432**

**Web: [www.brammerstandard.com](http://www.brammerstandard.com)**  
**Email: [contact@brammerstandard.com](mailto: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](http://www.brammerstandard.com)**

## **References:**

Versions used were those available at the time of testing and characterization

- E 826 Standard Practice for Testing Homogeneity of a Metal Lot or Batch in Solid Form by Spark Atomic Emission Spectrometry
- E 1019 Standard Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Fusion Techniques
- E 1806 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, 1916 Race Street, Philadelphia, PA, 19103.*

*ISO Guides and Standards available from Global Engineering - [www.global.ihs.com](http://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 May 7, 2013.

Beau R. Brammer