

BRAMMER STANDARD COMPANY, INC.

Certificate of Analysis

BS 101/3

Reference Material for Basic Oxygen Furnace Slag¹

	Certified Value ²	Estimate of Uncertainty ³	Certified Values ⁴	Certified Value ²	Estimate of Uncertainty ³
Al ₂ O ₃	1.42	0.08			
CaO	54.4	0.6			
Tot. Fe	10.9	0.6			
K ₂ O	0.005	0.001			
MgO	3.0	0.2			
MnO	5.0	0.1			
Na ₂ O	0.027	0.002			
P ₂ O ₅	0.74	0.03			
S	0.18	0.02			
SiO ₂	18.8	0.4			

Informational Values^{4,5}

TiO₂ (0.9)

¹ This certificate is a revision. For more information on the nature and extent of the revision, see the revision statement on page 5.

² 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.

³ 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.

⁴ Values are given in weight percent.

⁵ Values in parentheses are not certified and are provided for information only.

The requirements of ISO Guides 30, 31, and 35 were followed for the preparation of this Certified Reference Material and certificate of analysis.

BS 101/3

* Code for method

Certified values listed as weight percent

Analysis	*	Al ₂ O ₃	*	CaO	*	Tot. Fe	*	K ₂ O	*	MgO	*	MnO	*	Na ₂ O	*	P ₂ O ₅
1	17	1.13	17	53.2	4	9.811767	17	0.004	17	2.7	17	4.53	17	0.021	17	0.60
2	17	1.34	17	53.4	17	10.6	17	0.004	17	2.7	17	4.5	17	0.024	17	0.72
3	17	1.40	17	53.4	17	10.7	17	0.004	17	2.78	17	4.9	17	0.028	17	0.72
4	17	1.40	17	53.8	17	10.9	17	0.006	17	2.8	17	5.0	4	0.029	4	0.770533
5	4	1.501567	17	54.4	17	11.0	17	0.006	4	3.128067	17	5.0	17	0.029	17	0.88
6	17	1.53	17	54.5	17	11.4			17	3.2	17	5.0	17	0.040	17	0.90
7	17	1.68	17	54.74	17	11.5			17	3.2	17	5.0				
8	17	1.70	17	55.8	17	11.7			17	3.4	17	5.2				
9			4	56.15467					17	3.9	17	5.2				
10											4	5.2402				
11											17	5.43				
Average		1.417		54.38		10.89		0.00446		3.01		4.98		0.0275		0.737
Std dev		0.054		0.13		0.39		0.00020		0.10		0.15		0.0012		0.030
H		0.013		0.25		0.06		0.00058		0.02		0.03		0.0013		0.008
U ₁		0.056		0.28		0.39		0.00062		0.10		0.15		0.0018		0.031
t-statistic		2.36		2.31		2.36		2.78		2.31		2.23		2.57		2.57
U ₂		0.13		0.65		0.92		0.0017		0.24		0.34		0.0046		0.081
U ₃		0.046		0.22		0.33		0.00077		0.080		0.10		0.0019		0.033
Certified		1.42		54.4		10.9		0.005		3.0		5.0		0.027		0.74
Uncertainty		0.08		0.6		0.6		0.001		0.2		0.1		0.002		0.03
Tolerance		0.24		1.8		1.8		0.003		0.6		0.3		0.005		0.08

Analysis	*	S	*	SiO ₂
1	1	0.14	4	17.66383
2	1	0.14	17	17.80
3	1	0.17	17	17.8
4	1	0.19	17	18.6
5	1	0.20	17	18.83
6	1	0.22	17	19.2
7	1	0.22	17	19.2
8	1	0.24	17	19.54
9	1	0.253333	17	19.8
10			17	20.0
Average		0.1807		18.84
Std dev		0.0062		0.39
H		0.0035		0.10
U ₁		0.0071		0.40
t-statistic		2.31		2.26
U ₂		0.016		0.90
U ₃		0.0054		0.28
Certified		0.18		18.8
Uncertainty		0.02		0.4
Tolerance		0.06		1.2

Analysis	*	TiO ₂
1	17	0.55
2	17	0.70
3	17	0.87
4	4	0.9014
5	17	0.97
6	17	0.99
7	17	1.12
Average		0.87
Std dev		1.020
H		0.01
U ₁		1.020
t-statistic		2.45
U ₂		2.49
U ₃		0.94
(Informational)		(0.9)

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 it's 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 times 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.

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 Difference |
| 5 ICP Mass Spectrometry | 11 GD Atomic Emission | 17 Wet |
| 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>
Crobaugh Laboratories	Cleveland, OH		
Andrew S. McCreath & Son, Inc.	Harrisburg, PA		
Coors Spectro-Chemical Laboratory	Golden, CO		
Ledoux & Company	Teaneck, NJ		
Jones & Laughlin	Wadsworth, OH		
US Steel Research Laboratory	Monroeville, PA		
Carpenter Technology Corporation	Reading, PA		
Youngstown Steel & Tube Research	Youngstown, OH		
Brammer Standard Company, Inc.	Houston, TX		
Wheeling Pittsburgh Steel	Wheeling, WV		
Bethlehem Steel	Bethlehem, PA		
Youngstown Steel & Tube, Indiana Harbor	Valparaiso, IN		
Jones & Laughlin	Wadsworth, OH		
US Steel Research Laboratory	Monroeville, PA		
Laboratory Testing, Inc.	Hatfield, PA	PRI/Nadcap	17025

Nadcap = National Aerospace and Defense Contractors Accreditation Program

PRI =Performance Review Institute

Analysis: Chemical analyses were made on samples taken from bulk powder material. The original participating laboratories normally followed the requirements of ISO Guide 17025. The laboratories participating in the stability testing followed the requirements of ISO Standard 17025. Methods of analysis used were a those listed on pages 2-3.

Traceability: The following Reference Materials were used to validate the analytical data listed on pages 2-3 — LECO 502-265.

Homogeneity: This Reference Material (RM) 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 101/3 is valid for 20 years from the date of this certificate. The certification is nullified if this RM is damaged, contaminated, or otherwise modified.

Storage: This RM will remain stable for twenty years, provided that the bottle remains sealed and is stored in a cool, dry atmosphere. When the bottle has been opened, the lid should be secured immediately after use. If the contents should become discolored (eg. Oxidized) due to atmosphere contamination, they should be discarded.

Source: This material was produced by Jones & Laughlin Steel Plant; Aliquippa, PA. This material was made in a Basic Oxygen Furnace.

Form: This RM is available only in the form of minus 100 mesh powder in 100 gram bottles.

Use: This RM is intended for use in x-ray spectrometric and solution methods of analysis. Refer to ISO Guide 33 for information about the use of Reference Materials.

Sample Preparation: For best analytical results, use the same method for preparing the analytical surface on all reference materials as you use for production specimens.

Certificate Number: The unique identification number for this certificate of analysis is REV101/3-033017. You may obtain information on revisions of certificates from the internet at 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:

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**

Expiration: 20 years from date of certification, which is March 30, 2037. This material is valid until that date.

Revision: This reference material was originally certified as a reference material in September 1976. It was revised on April 20, 1989 to add Sodium and Potassium Oxides. It was revised on February 02, 2009 to add uncertainties. The documents validity for Brammer Standard powder products in 20 years. Additional inter-laboratory testing was performed in 2016 to prove stability and has been included on this certificate. TiO₂ has been changed from certified to informational. K₂O and Na₂O has been changed from informational to certified. Revised values for all elements except SiO₂ are presented.

Brammer Standard Company, Inc., is accredited by the American Association For Laboratory Accreditation (A2LA) to ISO Standard 17034 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:

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 30:2015 Terms and definitions used in connection with reference materials + 2008 amendment
- ISO Guide 31:2015 Reference materials - Contents of certificates and labels
- ISO Guide 33:2015 Uses of certified reference materials
- ISO Standard 17034:2016 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 Drive, West Conshohocken, PA 19428.

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

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

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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 March 30, 2017.

Beau R. Brammer

President