

Revised Certificate of Analysis

Reference Material for Carbon, Sulfur, Nitrogen, Oxygen, and Hydrogen in Stainless Steel

B.S. HON - 1

	Certified Value ₁	Estimate of Uncertainty _{2,3}
Carbon	0.049	±0.001
Sulfur	0.0025	±0.0003
Nitrogen	0.03627	±0.00072
Oxygen	0.00564	±0.00027
Hydrogen	0.00019	±0.00003

Values expressed as weight percent

Due to slight variations in pin weights, best precision and accuracy will be obtained by weighing each pin before analysis.

¹ The certified value listed is the present best estimate of the true value.

² The uncertainties listed for carbon and sulfur are based on the confidence interval formula:

$$c(95\%) = \frac{s_M \times t}{\sqrt{n}}$$

where: s_M = Standard Deviation of Laboratory Means
 t = Students t value
 n = number of laboratories

For further information regarding the confidence interval for the certified value, see ISO Guide 35:1989, section 4.

³ The uncertainties listed for nitrogen, oxygen, and hydrogen are based on value judgements of the material inhomogeneity and the standard deviation for each batch of analysis.

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. Inquires concerning this Reference Material should be directed to:

Brammer Standard Co., Inc.
14603 Benfer Road
Houston, Texas 77069 USA

Phone: (281) 440-9396

www.brammerstandard.com

Fax: (281) 440-4432

REVISION: This certificate of analysis is revised to assign a new value for hydrogen. It has been found that the nickel-plating of different batches of pins affects the hydrogen content of each batch. This certificate covers the second batch that was nickel-plated.

Certified by: G. R. Brammer _____ on April 7, 2005.

Description of the sample

This sample is available only in the form of pins with an approximate diameter of 3.8 mm (0.15 in.) and a length of 11.2 mm (0.44 in.). This reference material is sold in units of 250 one gram pins. The material used for making BS HON was AISI grade 302 stainless steel.

Source: The material for this reference material was melted and fabricated into wire by the Carpenter Technology Corporation in Reading, Pennsylvania. The wire was processed into pins by SKF Bearing Industries Company in Bremen, Indiana.

Homogeneity: This Reference Material was tested for homogeneity using ASTM Standard Practice E 826 and found acceptable.

Traceability for carbon and sulfur certified values: The final data for this reference material were calculated based on the values obtained from NIST SRMs 2166, 2167, and ECRM 292-1. This reference material is therefore directly traceable to NIST certified reference materials.

Use: This Reference Material is intended for use in combustion and fusion instrument methods of analysis.

Caution: Due to slight variations in pin weights, best precision and accuracy will be obtained by weighing each pin before analysis.

Sample Preparation: No special preparation of this reference material is necessary. The pins have been solution plated with tin and nickel.

Interlaboratory Testing Procedure

Each participating laboratory received a set of test samples consisting of the Brammer Reference Materials BS HON, plus three Certified Reference Materials (CRMs) as shown in the table on the following page. The CRMs were submitted as unknown samples to each laboratory in coded containers. The participating laboratories were instructed to calibrate their instruments by their normal procedures and to analyze the samples in duplicate on two different days. All instruments used were manufactured by the LECO Corporation. The models used are listed below.

Lab	Model	Lab	Model
1	CS-444	6	CS-444
2	CS-444	7	CS-344
3	CS-244	8	CS-444
4	CS-244	9	CS-344
5	CS-444	10	CS-444

Certified Values for carbon and sulfur BS HON

The laboratory means and standard deviations are listed on the following pages. The carbon and sulfur analyses were determined on calibrations that were validated with NIST CRMs and are therefore traceable to NIST. Regression calculations were used to produce the final certified carbon and sulfur values.

Participating laboratories were:

Allegheny Ludlum Steel Corp., Lockport, New York
P. S. Widmer

Brammer Standard Company, Houston, Texas
Richard Beaumont

Copperweld Steel Company, Warren, Ohio
A. J. Selak

Crucible Specialty Metals, Syracuse, New York
Chuck Merriam, Debbie Filip

LTV Steel Company, Indiana Harbor Works, E. Chicago, Indiana
John Hlebek

Lukens Steel Company, Coatesville, Pennsylvania
Sam Forese

Republic Engineered Steels, Canton, Ohio
Sherry Stroup

The Timken Company, Harrison Plant, Canton, Ohio
David Leatherbarrow

The Timken Company, Faircrest Steel Plant, Canton, Ohio
Debbie Montgomery

Vac Air Alloys Corporation, Frewsburg, New York
Michele Clendenen

Certified Values for nitrogen, oxygen, and nitrogen

Refer to pages 5 through 8 for nitrogen, oxygen, and nitrogen analysis information.

Lab Code	Day 1 Run 1	Run 2	Day 2 Run 1	Run 2	Lab Average	Within Lab 1s	Regression Results
1	0.0475	0.0480	0.0529	0.0468	0.04880	0.00278	0.04811
2	0.0452	0.0451	0.0451	0.0446	0.04500	0.00027	0.04842
3	0.0472	0.0480	0.0496	0.0488	0.04840	0.00103	0.04563
4	0.050	0.047	0.051	0.049	0.04925	0.00171	0.05020
5	0.0463	0.0471	0.0477	0.0480	0.04728	0.00075	0.04819
6	0.0500	0.0490	0.0500	0.0502	0.04980	0.00054	0.04909
7	0.04778	0.06167	0.04826	0.04784	0.05139	0.00686	0.05194
8	0.04829	0.04884	0.04945	0.05077	0.04934	0.00107	0.04873
9	0.0482	0.0483	0.0483	0.0477	0.04813	0.00029	0.04853
10	0.049375	0.048969	0.047929	0.049235	0.04888	0.00065	0.04688
					Average	0.04863	0.04857
					1s	0.00168	0.00171

NIST SRM 2166 Certified Carbon = 0.0150%, uncertainty = 0.001%

Lab Code	Day 1 Run 1	Run 2	Day 2 Run 1	Run 2	Lab Average	Within Lab 1s
1	0.0170	0.0217	0.0188	0.0196	0.01928	0.00195
2	0.0140	0.0144	0.0141	0.0141	0.01415	0.00017
3	0.0183	0.0192	0.0172	0.0212	0.01898	0.00169
4	0.015	0.014	0.012	0.013	0.0135	0.00129
5	0.0145	0.0143	0.0145	0.0150	0.01458	0.00030
6	0.0153	0.0151	0.0155	0.0150	0.01523	0.00022
7	0.01507	0.01495	0.01318	0.01302	0.01406	0.00111
8	0.01664	0.01643	0.01741	0.01754	0.01701	0.00055
9	0.0151	0.0152	0.0150	0.0150	0.01508	0.00010
10	0.014809	0.015281	0.014906	0.015282	0.01507	0.00025
					Average	0.01569
					1s	0.00204

NIST SRM 2167 Certified Carbon = 0.051%, uncertainty = 0.002%

Lab Code	Day 1 Run 1	Run 2	Day 2 Run 1	Run 2	Lab Average	Within Lab 1s
1	0.0548	0.0521	0.0502	0.0498	0.05173	0.00228
2	0.0475	0.0482	0.0474	0.0471	0.04755	0.00047
3	0.0520	0.0499	0.0542	0.0518	0.05198	0.00176
4	0.046	0.050	0.053	0.045	0.0485	0.00370
5	0.0497	0.0499	0.0503	0.0501	0.05000	0.00026
6	0.0516	0.0523	0.0520	0.0510	0.05173	0.00056
7	0.05038	0.05059	0.04941	0.04875	0.04978	0.00086
8	0.04908	0.04957	0.05256	0.05255	0.05094	0.00188
9	0.0505	0.0506	0.0506	0.0509	0.05065	0.00017
10	0.052006	0.052886	0.052572	0.052403	0.05247	0.00037
					Average	0.05053
					1s	0.00159

ECRM 292-1 Certified Carbon = 0.0367%, uncertainty = 0.0008%

Lab Code	Day 1 Run 1	Run 2	Day 2 Run 1	Run 2	Lab Average	Within Lab 1s
1	0.0375	0.0385	0.0393	0.0366	0.03798	0.00118
2	0.0338	0.0342	0.0337	0.0338	0.03388	0.00022
3	0.0390	0.0399	0.0413	0.0482	0.04210	0.00418
4	0.038	0.039	0.037	0.038	0.0380	0.00082
5	0.0356	0.0360	0.0363	0.0362	0.03603	0.00031
6	0.0374	0.0371	0.0375	0.0370	0.03725	0.00024
7	0.03646	0.03739	0.03580	0.03896	0.03715	0.00137
8	0.03712	0.03775	0.03999	0.04026	0.03878	0.00158
9	0.0364	0.0364	0.0362	0.0362	0.03630	0.00012
10	0.038015	0.038381	0.042774	0.03815	0.03933	0.00230
					Average	0.03768
					1s	0.00219

1s = one standard deviation

Lab Code	Day 1 Run 1	Run 2	Day 2 Run 1	Run 2	Lab Average	Within Lab 1s	Regression Results
1	0.0034	0.0035	0.0039	0.0033	0.00353	0.00026	0.00286
2	0.0023	0.0024	0.0023	0.0024	0.00235	0.00006	0.00237
3	0.0032	0.0032	0.0026	0.0027	0.00293	0.00032	0.00259
4	0.0032	0.0030	0.0029	0.0031	0.00305	0.00013	0.00290
5	0.0021	0.0024	0.0025	0.0025	0.00238	0.00019	0.00234
6	0.0023	0.0024	0.0022	0.0020	0.00223	0.00017	0.00221
7	0.00236	0.00229	0.00291	0.00285	0.00260	0.00032	0.00219
8	0.00335	0.00313	0.00244	0.00295	0.00297	0.00039	0.00274
9	0.0026	0.0030	0.0027	0.0025	0.00270	0.00022	0.00263
10	0.002241	0.002401	0.001884	0.001957	0.00212	0.00024	0.00201
				Average	0.00268	0.00023	0.00248
				1s	0.00044		0.00030

NIST SRM 2166 Certified Sulfur = 0.0023%, uncertainty = 0.0002%

Lab Code	Day 1 Run 1	Run 2	Day 2 Run 1	Run 2	Lab Average	Within Lab 1s
1	0.0023	0.0029	0.0034	0.0026	0.00280	0.00047
2	0.0024	0.0022	0.0022	0.0023	0.00228	0.00010
3	0.0027	0.0026	0.0024	0.0024	0.00253	0.00015
4	0.0026	0.0029	0.0017	0.0021	0.00233	0.00053
5	0.0021	0.0022	0.0023	0.0023	0.00223	0.00010
6	0.0022	0.0023	0.0024	0.0020	0.00223	0.00017
7	0.00259	0.00273	0.00268	0.00263	0.00266	0.00006
8	0.00246	0.00268	0.00184	0.00207	0.00226	0.00038
9	0.0023	0.0023	0.0023	0.0022	0.00228	0.00005
10	0.002568	0.002253	0.001996	0.002218	0.00226	0.00024
				Average	0.00238	0.00022
				1s	0.00020	

NIST SRM 2167 Certified Sulfur = 0.0091%, uncertainty = 0.0002%

Lab Code	Day 1 Run 1	Run 2	Day 2 Run 1	Run 2	Lab Average	Within Lab 1s
1	0.0096	0.0096	0.0098	0.0099	0.00973	0.00015
2	0.0089	0.0089	0.0088	0.0088	0.00885	0.00006
3	0.0092	0.0093	0.0094	0.0090	0.00923	0.00017
4	0.0090	0.0092	0.0095	0.0086	0.00908	0.00038
5	0.0088	0.0087	0.0090	0.0093	0.00895	0.00026
6	0.0088	0.0088	0.0090	0.0085	0.00878	0.00021
7	0.0114	0.0115	0.0102	0.0102	0.01083	0.00072
8	0.01120	0.01157	0.01087	0.01068	0.01108	0.00039
9	0.0086	0.0083	0.0085	0.0087	0.00853	0.00017
10	0.012495	0.013083	0.010739	0.010467	0.01170	0.00129
				Average	0.00967	0.00038
				1s	0.00112	

ECRM 292-1 Certified Sulfur = 0.0055%, uncertainty = 0.0002%

Lab Code	Day 1 Run 1	Run 2	Day 2 Run 1	Run 2	Lab Average	Within Lab 1s
1	0.0066	0.0064	0.0066	0.0066	0.00655	0.00010
2	0.0054	0.0054	0.0054	0.0054	0.00540	0.00000
3	0.0060	0.0061	0.0061	0.0060	0.00605	0.00006
4	0.0057	0.0061	0.0057	0.0061	0.00590	0.00023
5	0.0056	0.0057	0.0059	0.0058	0.00575	0.00013
6	0.0055	0.0057	0.0058	0.0053	0.00558	0.00022
7	0.00699	0.00640	0.00654	0.00694	0.00672	0.00029
8	0.00696	0.00687	0.00670	0.00675	0.00682	0.00012
9	0.0058	0.0057	0.0055	0.0054	0.00560	0.00018
10	0.007954	0.007860	0.008379	0.006388	0.00765	0.00087
				Average	0.00620	0.00022
				1s	0.00071	

1s = one standard deviation

OXYGEN, NITROGEN AND HYDROGEN ANALYSIS

SOME OF THE PARTICIPATING LABORATORIES

Allegheny Ludlum Steel Corp., Brackenridge, PA
Lawrence Electronics, Seattle, WA
LTV Steel Company, Canton, OH
Lukens Steel Company, Coatsville, PA
The Timken Company, Canton, OH
VacAir Alloys Corp., Frewsburg, NY

Analytical Methods

Each participating laboratory received a set of test specimens which consisted of the new Brammer Reference Material and other Reference Materials (RMs). The laboratories were requested to analyze the specimens in duplicate on two days. The data tables show all the analysis reported from each laboratory.

Hydrogen - All laboratories used instruments manufactured by LECO Corporation for the analysis with the exception of hydrogen analysis by laboratory number 5. Laboratory number 5 used a hot extraction and measurement system (HEMS) in which the sample is melted. The hydrogen testing instruments were calibrated by gas dosing and the calibration was verified with commercially available RMs. See page 6 for a listing of verification RMs. A 4 to 5 gram sample weight was used for each analysis.

Oxygen and Nitrogen - The calibration of the oxygen and nitrogen testing instruments were verified with a variety of commercially available RMs. Laboratories 2, 3, and 4 verified their nitrogen calibration with NIST SRM 73c Certified Reference Material.

Reference Materials

Reference Materials supplied for this test program were analyzed along with the BS HON to validate this certification procedure. The certified values of the RMs are listed along with the calculated average and standard deviation. The following RMs were used:

Number	Produced by	Certified for
CMSI 3016-1	China Metallurgical Standardization Research Institute	H
JSS GS-1c	The Iron and Steel Institute of Japan	O, N, H
NASAB 201S-1*	Nordisk Analys Service AB, Sweden	O, N
NASAB 301S-1*	Nordisk Analys Service AB, Sweden	O, N
502-061	LECO Corporation, USA	H

* The NASAB materials were formally sold with the prefix SKF.

Oxygen

Lab No.	Day 1		Day 2		Average	std dev	Overall		Certified Value
							Average	Std.Dev.	
NASAB 201S-1									
1	45.2	44.2	44.6	44.0	44.50	0.53			
2	41.0	41.0	39.0	42.0	40.75	1.26			
3	42.2	41.8	42.8	41.8	42.15	0.47			
4	40.0	39.0	40.0	43.0	40.50	1.73			
5	45.0	42.0	44.0	48.0	44.75	2.50			
6	43.0	41.0	41.0	40.0	41.25	1.26			
							42.32	2.13	44.9
NASAB 301S-1									
1	94.0	92.9	90.3	95.3	93.13	2.12			
2	94.0	96.0	94.0	93.0	94.25	1.26			
3	94.2	93.3	95.1	94.2	94.20	0.73			
4	92.0	89.0	88.0	91.0	90.00	1.83			
5	96.0	99.0	98.0	93.0	96.50	2.65			
							93.61	2.65	95.7
JSS GS-1c									
1	48.4	44.2	53.9	52.0	49.63	4.28			
2	48.0	48.0	46.0	46.0	47.00	1.15			
3	50.0	49.5	48.0	45.1	48.15	2.20			
4	47.0	43.0	46.0	42.0	44.50	2.38			
5	48.0	49.0	49.0	46.0	48.00	1.41			
6	49.0	46.0	44.0	43.0	45.50	2.65			
							47.13	2.80	46.0 ± 3.8
BS HON									
1	59.6	58.8	58.8	60.1	59.32	0.64			
2	57.0	57.0	57.0	56.0	56.75	0.50			
3	60.8	57.4	57.6	57.0	58.20	1.75			
4	57.0	54.0	52.0	55.0	54.50	2.08			
5	53.0	56.0	58.0	53.0	55.00	2.45			
6	57.0	54.0	54.0	53.0	54.50	1.73			
							56.38	2.38	

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Nitrogen

Lab No.	Day 1		Day 2		Average	std dev	Overall		Certified Value
							Average	Std.Dev.	
NASAB 201S-1									
1	434.0	441.0	438.0	419.0	433.00	9.76			
2	427.0	429.0	425.0	428.0	427.25	1.71			
3	429.0	432.2	424.8	427.9	428.48	3.05			
4	422.0	420.0	431.0	423.0	424.00	4.83			
5	428.0	432.0	426.0	429.0	428.75	2.50			
							428.29	5.38	428.9
NASAB 301S-1									
1	166.0	180.0	157.0	154.0	164.25	11.67			
2	180.0	178.0	178.0	177.0	178.25	1.26			
3	172.7	173.1	172.9	173.0	172.92	0.17			
4	158.0	157.0	166.0	164.0	161.25	4.43			
5	173.0	176.0	175.0	170.0	173.50	2.65			
							170.03	8.02	171.8
JSS GS-1c									
1	253.0	262.0	261.0	261.0	259.25	4.19			
2	256.0	253.0	255.0	252.0	254.00	1.83			
3	252.6	247.6	252.6	246.6	249.85	3.20			
4	256.0	252.0	255.0	257.0	255.00	2.16			
5	254.0	253.0	256.0	253.0	254.00	1.41			
6	257.0	260.0	268.0	258.0	260.75	4.99			
							255.47	4.57	254 ± 6
BS HON									
1	367.0	365.0	365.0	359.0	364.00	3.46			
2	366.0	360.0	359.0	357.0	360.50	3.87			
3	354.0	367.3	355.7	348.3	356.32	7.97			
4	367.0	366.0	357.0	360.0	362.50	4.80			
5	361.0	361.0	363.0	359.0	361.00	1.63			
6	375.0	367.0	375.0	371.0	372.00	3.83			
							362.72	6.25	

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Hydrogen ORIGINAL DATA NOVEMBER, 1989

Lab No.	Day 1	Day 2	Average	std dev	Overall Average	Std.Dev.	Certified Value
JSS GS-1c							
1	1.60	1.57	1.37	1.46			
2	1.36	1.36	1.53	1.47			
3	1.57	1.66	1.81	1.63			
4	1.64	1.51	1.74	1.73			
5	1.52	1.30	1.17	1.20	1.50	0.18	1.7 ± 0.2
CMSI 3016-1							
1	1.75	1.74	1.59	1.65			
2	2.06	2.10	1.84	2.06			
3	2.18	2.22	2.31	2.19			
4	2.18	2.17	2.42	2.26			
5	1.61	1.59	2.02	1.62	1.98	0.27	2.4 ± 0.25
502-061							
1	1.02	1.20	1.14	1.02			
2	1.05	1.19	1.12	1.13			
3	1.00	1.11	1.07	1.12			
4	1.09	1.09	1.32	1.12			
5	1.67	1.45	1.63	1.84	1.22	0.23	1.05 ± 0.22
Laboratory 5 data considered an outlier. Calculated after deleting Lab 5							
					1.11	0.08	
BS HON							
1	1.74	1.95	1.72	1.81			
2	2.14	2.01	2.22	2.19			
3	2.09	2.20	2.26	2.12			
4	2.10	2.33	2.34	2.30			
5	1.83	1.76	1.69	1.63	2.02	0.23	

Instruments used

Lab ID No.	Oxygen & Nitrogen Testing Instrument	Lab ID No.	Hydrogen Testing Instrument
1	TC 136, Mfg. by LECO Corporation	1	DH 103, Mfg. by LECO Corporation
2	TC 136	2	DH 103
3	TC 36 " " " " "	3	DH 103
4	TC 36	4	DH 103
5	TC 36	5	Hot extraction and measurement system
6	TC 136		

Test results of second nickel-plated batch, May, 2005

Check analysis of third batch of pins after plating.

Lab No.	1	2	3	Average	std dev	Overall Average	Std.Dev.	Certified Value
1	1.80	1.90	1.90	1.867	0.058			
2	1.84	1.76	1.70	1.767	0.070			
3	2.01	1.96	2.04	2.003	0.040	1.88	0.11	1.9

Certificate No. REV-HON-1-040705p8

Bias in Hydrogen Reference Materials

Recently, two different testing programs have been conducted to determine if a bias exists between the different sources of Reference Materials, (RMs), for hydrogen in steel. Both studies concluded that there is a bias between RMs produced by two USA sources and those produced by NASAB (formally SKF) in Sweden. The bias is approximately 0.2 $\mu\text{g/g}$ at the 1 $\mu\text{g/g}$ level and 1.5 $\mu\text{g/g}$ at the 6 $\mu\text{g/g}$ level. The RM suppliers indicate that the instruments were calibrated in the same manner - by gas dosing.

The BS HON Reference Material should compare well against RMs supplied by LECO Corporation since most laboratories verified their calibration with LECO RMs. A small bias may be observed if the BS HON is compared with NASAB and other Certified Reference Materials produced outside of the U.S.A..

NIST (formally NBS) does not currently supply Certified Reference Materials (CRMs) for hydrogen in steel. Requests have been made to NIST for hydrogen in steel CRMs and it is hoped that they can be supplied in the near future. After NIST produces a CRM for hydrogen in steel or if another accurate instrument for measuring hydrogen in steel is widely available, the BS HON will be retested.

Additional calibration verification data

Most participating laboratories reported that RMs in addition to those provided for this testing program were used to verify their calibration. This was performed each day immediately before the set of test specimens was analyzed. This additional data provided by the laboratories is listed below.

Lab No.	Reference Material	Reported Verification Data		
		Certified	Day 1	Day 2
1	LECO Corp. 502-061	1.05	1.08	1.12
2	LECO Corp. 501-529	5.6	5.63	5.69
	LECO Corp. 762-747	0.9	0.94	0.91
3	LECO Corp. 502-061	0.99		
	AISI AH-1	1.6		
	LECO Corp. 505-060	5.68		
4	LECO Corp. 502-060	5.40	5.38	5.33
	LECO Corp. 502-061	1.04	0.89	1.13
5	LECO Corp. Reference Materials NIST Titanium Certified Reference Material			

Certificate No. REV-HON-1-040705p9