

## National Institute of Standards & Technology

# Certificate of Analysis

## Standard Reference Material® 1763a

### Low Alloy Steel

(In Cooperation with ASTM International)

This Standard Reference Material (SRM) is a special low alloy steel. SRM 1763a is intended for use in the evaluation of chemical and instrumental methods of analysis and in calibration of analysis. Each unit of SRM 1763a consists of a disk approximately 34 mm in diameter and 19 mm thick.

Certified values for 17 constituents for SRM 1763a are reported in Table 1. Reference values for four constituents are reported in Table 2. Information values for two constituents are reported in Table 3. For all elements, values are reported as mass fractions [1]. Value assignment categories are based on the definition of terms and modes used at NIST for chemical reference materials [2].

**Certified Values:** A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or taken into account [1]. A certified value is the best estimate of the true value based on the results of analyses performed at NIST and collaborating laboratories using instrumental and classical test methods.

**Reference Values:** Reference values are non-certified values that are the best estimates of the true values. However, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may not include all sources of uncertainty.

**Information Values:** An information value is considered to be a value that will be of use to the SRM user, but insufficient information is available to assess the uncertainty associated with the value.

**Expiration of Certification:** The certification of this SRM is valid indefinitely, within the uncertainty specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate (see Instructions for Use). However, the certification will be nullified if the SRM is damaged or contaminated.

**Maintenance of SRM Certification:** NIST will monitor this material over the period of its certification. If substantive technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

Coordination of the technical measurements for certification of SRM 1763a was accomplished under the direction of A.F. Marlow of the NIST Analytical Chemistry Division.

Measurements for homogeneity testing and value assignment of SRM 1763a were performed at NIST by J.R. Sieber and A.F. Marlow of the NIST Analytical Chemistry Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

Stephen A. Wise, Chief Analytical Chemistry Division

Robert L. Watters, Jr., Chief Measurement Services Division

Gaithersburg, MD 20899 Certificate Issue Date: 08 July 2010 See Certificate Revision History on Last Page

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Statistical consultation for the value assignment of SRM 1763a was provided by J.H. Yen of the NIST Statistical Engineering Division.

#### INSTRUCTIONS FOR USE

The test surface is the side opposite to the labeled surface, which includes the SRM number. The entire thickness of the unit is certified. However, the user is cautioned not to measure disks less than 2 mm thick when using X-ray fluorescence spectrometry. Each packaged disk has been prepared by finishing the test surface using a milling machine. The user must determine the correct surface preparation procedure for each analytical technique. The user is cautioned to use care when either resurfacing the disk or performing additional polishing as these processes may contaminate the surface. It was found by NIST that abrasive paper must be changed frequently during surface grinding. Used paper loses its ability to remove contaminants from the surface of the steel. When not in use, the material should be stored in its original container in a cool, dry location. This material was tested using both the solid disks and chips prepared from the disks. The certified values are considered to be representative of the overall average composition of the material.

Table 1. Certified Values for SRM 1763a

Constituent	Mas	ss Frac (%)	tion <sup>(a)</sup>
Al	0.0435	±	0.0032
As	0.055	±	0.003
В	0.0054	±	0.0009
C	0.202	±	0.007
Co	0.093	$\pm$	0.003
Cr	0.498	±	0.007
Cu	0.042	$\pm$	0.001
Mn	1.584	$\pm$	0.021
Mo	0.490	±	0.011
Nb	0.100	$\pm$	0.005
Ni	0.513	$\pm$	0.012
P	0.0123	$\pm$	0.0015
S	0.022	$\pm$	0.002
Si	0.633	±	0.016
Ti	0.308	$\pm$	0.009
V	0.307	$\pm$	0.006
Zr	0.044	$\pm$	0.004

<sup>&</sup>lt;sup>(a)</sup> The uncertainty of a certified value is expressed as an expanded uncertainty, U, and is calculated according to the method described in the ISO Guide and NIST Guidelines [3]. The expanded uncertainty is  $U = ku_c$  where  $u_c$  is calculated, at the level of one standard deviation, by combining a between-method variance, a pooled within-method variance and a variance representing the uncertainty of the comparison of SRM 1763 and SRM 1763a. The coverage factor k = 2, was determined from the Student's t-distribution corresponding to the appropriate degrees of freedom and approximately 95 % confidence [3].

Table 2. Reference Values for SRM 1763a

Constituent	Mass Fraction <sup>(b)</sup> (%)		
N	0.0045	±	0.0007
Sb	0.011	±	0.002
Sn	0.011	$\pm$	0.003
Ta	0.012	$\pm$	0.002

<sup>&</sup>lt;sup>(b)</sup> The uncertainty of a reference value is expressed as an expanded uncertainty, U, and is calculated according to the method described in the ISO Guide and NIST Guidelines [3]. The expanded uncertainty is  $U = ku_c$  where  $u_c$  is calculated, at the level of one standard deviation, by combining a between-method variance, a pooled within-method variance and a variance representing the uncertainty of the comparison of SRM 1763 and SRM 1763a. The coverage factor k = 2, was determined from the Student's t-distribution corresponding to the appropriate degrees of freedom and approximately 95 % confidence [3].

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Table 3. Information Values for SRM 1763a

Constituent	Mass Fraction		
	(%)		
Fe	95.3		
W	0.002		

#### PREPARATION AND ANALYSIS<sup>(1)</sup>

The value assignments for SRM 1763a are based on a complete, high-precision comparison of this SRM with the original SRM 1763. The materials for both SRM 1763 and SRM 1763a come from the same batch of steel prepared in 1989. The two lots were shown to be indistinguishable for all elements, except C, N, and V. The value assignments for SRM 1763a are considered to be directly traceable to the primary reference materials and calibrations used for value assignment of SRM 1763. The value assignments for C and N were obtained by combustion with infrared detection using a calibration based on SRM 1763. The value assigned for V was obtained by X-ray fluorescence spectrometry using a calibration based on low alloy steel SRMs 1262 through 1265 and SRMs 1761 through 1768. The test methods employed in value assignment are listed in this certificate. High-precision measurements for homogeneity testing of SRM 1763a and high-precision comparisons of SRM 1763 and SRM 1763a were performed at NIST by X-ray fluorescence spectrometry and at Laboratory Testing, Inc. using inductively coupled plasma optical emission spectrometry and combustion with infrared detection.

The material for SRM 1763 and SRM 1763a was vacuum induction melted followed by vacuum arc re-melting at the Carpenter Technology Corporation, Reading, PA. The ingots were processed by Carpenter Technology to provide a material of low heterogeneity.

In 1989, the technical and support aspects involved in the original preparation, certification, and issuance of SRM 1763 were coordinated through the NIST Standard Reference Materials Program by P. A. Lundberg. The overall coordination of the technical measurements leading to certification of SRM 1763 was performed under the direction of J.I. Shultz, Research Associate (retired), ASTM/NIST Research Associate Program.

Measurements of the original SRM 1763 were made at NIST by D.E. Brown, R.W. Burke, L.E. Creasy, W.F. Koch, A.F. Marlow, J.A. Norris, P.A. Pella, M.V. Smith, T.W. Vetter, Xie Guirong, and Xu Fu Zheng of the NIST Inorganic Analytical Research Division.

#### **Cooperating Laboratories**

For comparison of SRM 1763 and SRM 1763a, determinations of B, C and N were performed by Laboratory Testing, Inc., Hatfield, PA.; L. Dilks

Analytical determinations for certification of the original SRM 1763 were performed by the following laboratories: Amax Research and Development Center, Golden, CO; R.C. Binns

American Cast Iron Pipe Company, Birmingham, AL; R.N. Smith, D.R. Denney, C.E. Meads, R.J. Huffman; J.M. Hudson, R. G. Moffett

ARMCO Research and Technology, Middletown, OH; C.C. Borland, M.D. Kaehler, J.W. Leeker, T.M. Minor, G.D. Smith, R.L. Swigert, H.P. Vail, S.B. Warman, B.J. Young

Carpenter Technology Corporation, Carpenter Steel Division, Reading, PA; T.R. Dulski

Central Bureau for Nuclear Measurements, Geel, Belgium; A. Lamberty, L. Van Nevel, P. DeBievre

The Timken Company, Canton, OH; N.J. Stecyk

Data for nitrogen was provided by AISI Technical Committee on Chemical Analysis courtesy of D.E. Gillum, ARMCO Research and Technology

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<sup>(1)</sup> Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

#### Test Methods Employed at NIST and the Cooperating Laboratories

Atomic absorption spectrophotometry: Al, As, Co, Cr, Cu, Mn, Mo, Sn Ti, V, Zr

Combustion with infrared detection: C, N, S

Direct current plasma optical emission spectrometry: B, Co, Mo, Nb, Sn, Ti, Zr

Gravimetry: C, Mn, P, Si

Inductively coupled plasma optical emission Spectrometry: Al, As, B, Co, Cu, Mo, Mn, Ni, P, Sn, Ta Ti, V, Zr

Photometry: Cr, Cu, Mn, Mo, P, Ti

Spark source optical emission spectrometry: Al, As, B, C, Co, Cr, Cu, Mn, Mo, Nb, Ni, P, S, Si, Sn, Ta, Ti, V, Zr

Titrimetry: Cr, Mn, Ni, S

X-ray fluorescence spectrometry: Al, As, Co, Cr, Cu, Mn, Mo, Nb, Ni, P, S Sb, Si, Sn, Ta, Ti, V, W, Zr

#### REFERENCES

- [1] Thompson, A.; Taylor, B.N.; Guide for the Use of the International System of Units (SI), NIST Special Publication 811, 2008 ed. (2008); available at http://physics.nist.gov/Pubs/ (accessed June 2010).
- [2] May, W.; Parris, R.; Beck II, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definition of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Special Publication 260-136 (2000); available at http://ts.nist.gov/MeasurementServices/ReferenceMaterials/PUBLICATIONS.cfm (accessed June 2010).
- [3] Guide to the Expression of Uncertainty in Measurement; ISBN 92-67-10188-9, 1st ed. ISO, Geneva, Switzerland (1993); see also Taylor, B.N.; Kuyatt, C.E.; Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results; NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC (1994); available at http://physics.nist.gov/Pubs/ (accessed June 2010).

Certificate Revision History: 08 July 2010 (This revision includes a correction to the uncertainty value for carbon listed in Table 1 and minor editorial changes.); 8 December 2008 (Original certificate date)

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program: telephone (301) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

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