National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 1761a

Low Alloy Steel

(In Cooperation with ASTM International)

This Standard Reference Material (SRM) is intended for use in the evaluation of chemical and instrumental methods of analysis and in calibration of instrumental methods of analysis. Each unit of SRM 1761a consists of a disk approximately 34 mm in diameter and 19 mm thick.

Certified values for 14 constituents for SRM 1761a are reported in Table 1. Reference values for seven constituents are reported in Table 2. An Information value for Fe is 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 the present best estimate of the true value for which NIST has the highest confidence in its accuracy, in that all known or suspected sources of bias have been investigated or accounted for by NIST. For this SRM, certified values are 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 present 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.

The value assignments for SRM 1761a are based on a complete, high-precision comparison of this SRM with the original SRM 1761. The material for SRM 1761a was shown to be indistinguishable from the original material for all elements, except C, N, and V. The value assignments for SRM 1761a are considered to be directly traceable to the primary reference materials and calibrations used for value assignment of SRM 1761. The value assignments for C and N were obtained by combustion and infrared detection using a calibration based on SRM 1761. The value assigned for V was obtained by X-ray fluorescence spectrometry using a calibration based on low alloy steel SRMs 1761 through 1768. The test methods employed in value assignment are listed in this certificate.

The uncertainty listed with each assigned value is an expanded uncertainty approximating a 95 % level of confidence [3] and is calculated according to the method in the ISO and NIST Guides [4]. The uncertainty of the comparison between SRM 1761 and SRM 1761a has been accounted for in the reported uncertainty estimates.

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"). The certification is nullified if the SRM is damaged, contaminated or otherwise modified.

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

Coordination of the technical measurements for certification of SRM 1761a was accomplished under the direction of A.F. Marlow of the NIST Analytical Chemistry Division.

Stephen A. Wise, Chief Analytical Chemistry Division

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

Gaithersburg, MD 20899 Certificate Issue Date: 29 February 2008

SRM 1761a

Statistical consultation for the value assignment of SRM 1761a was provided by J.H. Yen of the NIST Statistical Engineering Division.

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

Constituent	Mass Fraction (%)		
В	0.0023	±	0.0006
Al	0.055	±	0.005
Si	0.182	±	0.011
Р	0.042	\pm	0.002
S	0.037	±	0.006
Ti	0.173	\pm	0.007
V	0.054	±	0.002
Cr	0.222	±	0.006
Mn	0.679	\pm	0.010
Ni	1.981	±	0.019
Cu	0.298	±	0.006
Zr	0.012	±	0.002
Nb	0.021	±	0.002
Мо	0.103	±	0.002

Table 1. Certified Values for SRM 1761a

Table 2. Reference Values for SRM 1761a

Constituent	Mas	s Frac (%)	ction
С	1.05	±	0.02
Ν	0.0042	±	0.0002
Со	0.027	±	0.003
As	0.011	±	0.002
Sn	0.050	±	0.004
Sb	0.0052	±	0.0003
Та	0.050	±	0.006

Constituent	Mass Fraction (%)
Fe	95.0

PLANNING, PREPARATION, TESTING, AND ANALYSIS¹

The material for SRM 1761a 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 1761 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 1761 was performed under the direction of J.I. Shultz, Research Associate (retired), ASTM/NIST Research Associate Program.

Measurements of the original SRM 1761 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.

High-precision X-ray fluorescence measurements for homogeneity testing of SRM 1761a and high-precision comparisons of SRM 1761 and SRM 1761a were performed at NIST by J.R. Sieber and A.F. Marlow of the NIST Analytical Chemistry Division.

Cooperating Laboratories

For comparison of SRM 1761 and SRM 1761a, determinations of B using inductively coupled plasma optical emission spectrometry and of C and N using combustion with infrared detection were performed by L. Dilks, Laboratory Testing, Inc., Hatfield, PA.

Analytical determinations for certification of the original SRM 1761 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, D.E. Gillum, ARMCO Research and Technology

Test Methods Employed at NIST and the Cooperating Laboratories

Atomic Absorption Spectrophotometry: Al, Ti, Cr, V, Mn, Co, Cu, As, Zr, Mo, Sn

Combustion with Infrared Detection: C, N, S

Direct Coupled Plasma Optical Emission Spectrometry: B, Ti, Co, Zr, Nb, Mo, Sn

¹ 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.

Gravimetry: C, Si, P, Mn

Inductively Coupled Plasma Optical Emission Spectrometry: B, Al, P, Ti, Cr, V, Mn, Co, Ni, Cu, As, Zr, Nb, Mo,

Sn, Ta

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

Spark Source Optical Emission Spectrometry: B, C, Al, Si, P, S, Ti, Cr, V, Mn, Co, Ni, Cu, As, Zr, Nb, Mo, Sn, Ta

Titrimetry: S, Cr, Mn, Ni

X-Ray Fluorescence Spectrometry: Al, Si, P, S, Ti, Cr, V, Mn, Co, Ni, Cu, As, Zr, Nb, Mo, Sn, Sb, Ta

REFERENCES

- [1] Taylor, B.N.; *Guide for the Use of the International System of Units (SI)*; NIST Special Publication 811, U.S. Government Printing Office: Washington, DC (1995).
- [2] May, W.E.; Parris, R.M.; Beck II, C.M.; Fassett, J.D.; Greenberg, R.R.; Guenther, F.R.; Kramer, G.W.; Wise, S.A.; Gills, T.E.; Colbert, J.C.; Gettings, R.J.; MacDonald, B.S.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements*; NIST Spec. Pub. 260-136, p. 16, U.S. Government Printing Office, Washington, DC(2000).
- [3] Hahn, G.J.; Meeker, W.Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc., New York (1991).
- [4] ISO; Guide to the Expression of Uncertainty in Measurement; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: 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 <u>http://physics.nist.gov/Pubs/</u>.

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <u>http://www.nist.gov/srm</u>.