National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 1764a

Low Alloy Steel

This Standard Reference Material (SRM) is a low alloy steel and is intended for use in the evaluation of chemical and instrumental methods of analysis and in calibration of those methods. A unit of SRM 1764a consists of a disk approximately 34 mm in diameter and 19 mm thick.

Certified Values: Certified values for 15 constituents of SRM 1764a are reported in Table 1. 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-composition reference materials [2]. 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. A certified value is the present 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 for five constituents are reported in Table 2. Reference values are non-certified values that are the best estimates of the true values [2]. 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: Information values for two constituents are reported in Table 3. 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 [2].

Expiration of Certification: The certification of **SRM 1764a** 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, 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 notification.

Coordination of the technical measurements for certification of SRM 1764a was accomplished under the direction of A.F. Marlow of the NIST Analytical Chemistry Division.

Measurements for homogeneity testing and value assignment of SRM 1764a were performed at NIST by J.R. Sieber and A.F. Marlow of the NIST Analytical Chemistry Division.

Statistical consultation for the value assignment of SRM 1764a 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.

Stephen A. Wise, Chief Analytical Chemistry Division

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

Gaithersburg, MD 20899 Certificate Issue Date: 09 June 2009 The value assignments for SRM 1764a are based on comparison of this SRM with the original SRM 1764. In 1989, the technical and support aspects involved in the original preparation, certification, and issuance of SRM 1764 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 1764 was performed under the direction of J.I. Shultz, Research Associate, ASTM/NIST Research Associate Program [3].

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.

PREPARATION AND ANALYSIS¹

The value assignments for SRM 1764a are based on comparison of this SRM with the original SRM 1764. The materials for both SRM 1764 and SRM 1764a came from the same batch of steel prepared in 1989. The two lots were shown to be indistinguishable. The value assignments for SRM 1764a are considered to be directly traceable to the primary reference materials and calibrations used for value assignment of SRM 1764. The test methods employed in value assignment are listed in the Appendix to this certificate. Measurements for homogeneity testing of SRM 1764a and comparison of SRM 1764 and SRM 1764a were performed at NIST by X-ray fluorescence spectrometry and at Laboratory Testing, Inc., Hatfield, PA using inductively coupled plasma optical emission spectrometry and combustion with infrared detection.

The material for SRM 1764 and SRM 1764a 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 high homogeneity.

¹ Certain commercial equipment, instruments or materials are identified in this certificate to adequately specify 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.

Table 1. Certified Values for SRM 1764a

Constituent	Mass Fraction ^(a) (%)		
Al	0.0098	±	0.0015
As	0.0100	\pm	0.0021
С	0.592	±	0.017
Cr	1.468	±	0.031
Cu	0.5178	\pm	0.0063
Mn	1.193	\pm	0.058
Мо	0.2007	±	0.0051
Nb	0.0416	\pm	0.0024
Ni	0.2006	\pm	0.0045
Р	0.0210	\pm	0.0016
S	0.0118	±	0.0030
Si	0.0595	\pm	0.0036
Та	0.0297	\pm	0.0042
Ti	0.0286	\pm	0.0023
V	0.1063	\pm	0.0021

^(a) The uncertainty of each certified value is expressed as an expanded uncertainty, U, and is calculated according to the method described in the ISO Guide [4]. The expanded uncertainty is calculated as $U = ku_c$, 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 1764 and SRM 1764a. The coverage factor, k = 2, was chosen to approximate a 95 % confidence level [5].

Constituent	Mass	Mass Fraction ^(b) (%)	
		(/0)	
В	0.0010	±	0.0003
Co	0.012	±	0.005
Ν	0.0023	\pm	0.0005
Sn	0.024	±	0.008
Zr	0.0012	\pm	0.0005

^(b) The uncertainty of each reference value is expressed as an expanded uncertainty, U, and is calculated according to the method described in the ISO Guide [4]. The expanded uncertainty is calculated as $U = ku_c$, 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 1764 and SRM 1764a. The coverage factor, k = 2, was chosen to approximate a 95 % confidence level [5].

Table 3. Information Values for SRM 1764a

Constituent	Mass Fraction
	(%)
Fe	95.1
W	0.0016

- [1] Thompson A., 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 (2008); available at http://ts.nist.gov/WeightsAndMeasures/Metric/mpo_pubs.cfm.
- [2] May, W.; Parris, R.; Beck, C.; Fassett, J.; Greenberg, R.; Guenther, F.; Kramer, G.; Wise, S.; Gills, T.; Colbert, J.; Gettings, R.; MacDonald, B.; *Definitions of Terms and Modes Used at NIST for Value-Assignment of Reference Materials for Chemical Measurements;* NIST Special Publication 260-136, U.S. Government Printing Office, Gaithersburg, MD (2000); available at http://www.cstl.nist.gov/nist839/NIST_special_publications.htm.
- [3] SRM 1764; *Low Alloy Steel*; National Institute of Standards and Technology; U.S. Department of Commerce: Gaithersburg, MD (26 February 1993).
- [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>.
- [5] Hahn, G.J.; Meeker, W.Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc., New York (1991).

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) 975-2200; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet at <u>http://www.nist.gov/srm.</u>

APPENDIX A

Collaborating Laboratories

For comparison of SRM 1764 and SRM 1764a, determinations of B, C, N and S were performed by Laboratory Testing, Inc., Hatfield, PA.; L. Dilks

Analytical determinations for certification of the original SRM 1764 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

The Timken Company, Canton, OH; N.J. Stecyk, D. Gapen, G. Hanni, L. McFarland, M. Moffat

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

Test Methods Employed at NIST and the Collaborating Laboratories

Atomic absorption spectrophotometry:	Al, Ti, Cr, V, Mn, Co, Ni, Cu, As, Zr, Mo, Sn
Combustion with infrared detection:	C, N, S
Direct current plasma optical emission spectrometry:	B, Ti, Co, Zr, Nb, Mo, Sn
Gravimetry:	C, Si, P, Mn, Ni
Inductively coupled plasma optical emission spectrometry:	B, Al, P, Ti, Cr, V, Mn, Co, Ni, Cu, As, Zr, Nb, Mo,
	Sn, Ta
Isotope dilution mass spectrometry:	S
Spectrophotometry:	B, 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:	P, S, Cr, Mn, Ni
X-ray fluorescence spectrometry:	Al, Si, P, S, Ti, V, Cr, Mn, Co, Ni, Cu, As, Zr, Nb,
	Mo, Sn, Ta, W