National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 125b

Low-Alloy High-Silicon Steel

This Standard Reference Material (SRM) is a low-alloy steel issued in the annealed condition and containing a high amount of silicon. SRM 125b is intended for use in the evaluation or calibration of chemical and instrumental methods of analysis. A unit of SRM 125b consists of a bottle containing approximately 100 g of chips.

Certified Mass Fraction Values: Certified values for 10 constituents of SRM 125b are reported in Table 1 as mass fractions [1]. Value assignment categories are based on the definitions of terms and modes used at NIST for chemical 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.

Reference Mass Fraction Values: A reference value for one constituent is reported in Table 2. A reference value is a non-certified value that is the best estimate of the true value; however, the value does not meet the NIST criteria for certification and is provided with the associated uncertainty that may not include all sources of uncertainty [2].

Information Mass Fraction Values: Information values for six constituents are reported in Table 3. An information value is considered to be a value that will be of interest to the SRM user, but insufficient information is available to assess the uncertainty associated with the value.

Expiration of Certification: The certification of **SRM 125b** 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"). Accordingly, periodic recalibration or recertification of this SRM is not required. The certification is nullified if the SRM is damaged, contaminated or otherwise modified.

Maintenance of SRM Certification: NIST will monitor this material 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 original certification of SRM 125b in 1970 was performed by O. Menis of the former Office of Standard Reference Materials of the National Bureau of Standards and J.I. Schultz, ASTM Research Associate. Coordination of the reevaluation of SRM 125b was performed by J.R. Sieber of the NIST Analytical Chemistry Division.

Measurements for value assignment of SRM 125b were performed at NIST by J.R. Baldwin, D.D. Bendigo, E.R. Deardorff, E.L. Garner, A.F. Marlow, T.J. Murphy, T.C. Rains, T.A. Rush, J.R. Sieber, B.A. Thompson, and S.A. Wicks of the NIST Analytical Chemistry Division. Homogeneity testing at NIST was performed by J.R. Baldwin, D.M. Bouchette, R.F. Brady, J. McKay, S.D. Rasberry, J.L. Webber, and S.A. Wicks. Analyses were also performed by M. Dannis and R.L. LeRoy, Armco Steel Corporation, Middletown, OH, and L.M. Melnick, W.R. Bandi, H.S. Karp, J.L. Lutz, R.W. Lewis, J.B. Ferons, and H.M. Lewis, United States Steel Corporation, Monroeville, PA.

Statistical consultation for the value assignment of SRM 125b was provided by J.H. Yen of the NIST Statistical Engineering Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 14 April 2011
Certificate Revision History on Last Page

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

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

INSTRUCTIONS FOR USE

To relate analytical determinations to the certified values on this Certificate of Analysis, a minimum sample quantity of 250 mg is recommended. When not in use, the material should be stored in its original container in a cool, dry location.

PREPARATION AND ANALYSIS[1]

The material for SRM 125b was prepared by the Armco Steel Corporation (Middletown, Ohio). A single ingot was pressed to a slab after having one dimension of the cross section four times that of the other dimension. After cropping top and bottom, the slab was cut lengthwise and the center section, corresponding to about one-fourth of the original ingot, was discarded. Slab sections were hot rolled to oversized rods and centerless ground to final rod size of 31.8 mm diameter. Some rods were chipped to prepare SRM 125b.

Homogeneity testing was performed at NIST and included metallographic studies, optical emission spectrometry, X-ray fluorescence spectrometry, and chemical analysis. The testing revealed the material to be homogeneous.

Certified Value Assignment: Each certified value is the mean of the method means available for that analyte. A method mean may combine the results of closely related methods. The uncertainty provided with each value is an expanded uncertainty about the mean to cover the measurand with approximately 95 % confidence. The expanded uncertainty is calculated as $U = ku_c$, where u_c incorporates both the observed differences among the results from the methods and their respective uncertainties, consistent with the ISO Guide and Supplement 1, and k is a coverage factor corresponding to approximately 95 % confidence for each analyte [3–5]. Analytical test methods are shown in Table 4.

Table 1. Certified Mass Fraction Values for SRM 125b

Constituent	Mass Fraction (%)	Expansion Factor, k
C	0.0261 ± 0.0030	3.2
Cr	0.0198 ± 0.0017	2.0
Cu	0.0707 ± 0.0011	2.0
Mn	0.2751 ± 0.0037	2.0
Mo	0.0087 ± 0.0007	2.0
Ni	0.0375 ± 0.0011	2.0
P	0.0276 ± 0.0012	2.8
S	0.0095 ± 0.0022	2.8
Si	2.889 ± 0.018	2.0
Sn	0.0034 ± 0.0004	2.0

Reference Value Assignment: The reference value is the unweighted mean of three method means. The uncertainty of the reference value is expressed as an expanded uncertainty, U, and is calculated according to the method described in the ISO Guide [3]. The expanded uncertainty is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined effect of estimated between-method and within-method components of uncertainty [3]. Analytical test methods are shown in Table 4.

Table 2. Reference Mass Fraction Value for SRM 125b

Constituent	Mass Fraction (%)	Expansion Factor, k
Al	0.329 ± 0.007	4.3

[1]Certain commercial equipment, instrumentation, or materials are identified in this certificate to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by NIST, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

Table 3. Information Mass Fraction Values for SRM 125b

Constituent	Mass Fraction (%)
As	0.003
Co	0.007
Fe	95.8
Sb	0.0009
Ti	0.004
V	0.0005
Zr	0.001

Table 4. Analytical Methods for SRM 125b

Element	Method(s)[(a)]	Element	Method(s)[(a)]
Al	1, 2, 3	Ni	4, 16
As	4	P	4, 17, 18
C	5, 6	S	4, 19
Co	4	Sb	4
Cr	4, 7, 8, 9	Si	4, 20, 21
Cu	4, 9, 10, 11	Sn	4, 22, 23
Fe	4	Ti	4
Mn	4, 12, 13, 14	V	4
Mo	4, 7, 15	Zr	4

[(a)]Key to Methods in Table 4:

1. Flame emission spectrometry
2. Precipitation as phosphate, ignition and weighing as $AlPO_4$
3. Ether, Hg cathode, cupferron, chrome-azurol-S spectrometric method
4. X-ray fluorescence spectrometry
5. Direct combustion followed by gravimetric determination
6. Direct combustion followed by thermal conductivity detection
7. Neutron activation analysis
8. Oxidation with peroxydisulfate followed by titration with $KMnO_4$
9. Diphenylcarbazide spectrometric method
10. Diethyldithiocarbamate spectrometric method
11. Isotope dilution mass spectrometry
12. Peroxydisulfate arsenite method
13. Potentiometric titration
14. Periodate spectrometric method
15. Sulfide-iodine method [6]
16. Photometric method
17. Spectrophotometric molybdenum-blue method
18. Ammonium phosphomolybdate extraction with isobutyl alcohol, reduced to molybdenum blue, and determined spectrometrically
19. Combustion and absorption in starch-iodide solution followed by titration with KIO_3 solution
20. Gravimetric determination after $HClO_4$ dehydration
21. Double dehydration with intervening filtration
22. Polarographic determination
23. Thiocyanate-ethylene glycol molybdenum complex extracted with monobutyl ether and determined spectrophotometrically

REFERENCES

[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://www.nist.gov/pml/pubs/index.cfm/ (accessed Apr 2011).

[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.nist.gov/srm/publications.cfm (accessed Apr 2011).

[3] JCGM 100:2008; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Apr 2011); 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 Apr 2011).

[4] JCGM 101:2008; *Evaluation of measurement data – Supplement 1 to the Guide to the Expression of Uncertainty in Measurement – Propagation of Distributions Using a Monte Carlo Method*; Joint Committee for Guides in Metrology (JCGM) (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_101_2008_E.pdf (accessed Apr 2011).

[5] Efron, B.; Tibshirani, R.J.; *An Introduction to the Bootstrap*; Chapman & Hall, London, UK (1993).

[6] J. Res. Nat. Bur. Stand. Vol. 8, p. 309 (1932) RP415.

Certificate Revision History: 14 April 2011 (This revision updates the certificate to current NIST standards and reports revised constituents and values); 16 October 1995 (editorial revision to include program and organizational changes at NIST); 24 February 1982 (revision of certificate to include a value for calcium); 10 April 1970 (original certificate date).

Users of this SRM should ensure that the Certificate of Analysis 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.