

National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 1755

Nitrogen in Low Alloy Steel (In Cooperation with the American Society for Testing and Materials)

This Standard Reference Material (SRM) is intended for use primarily in optical emission spectrometric methods of analysis. A unit of SRM 1755 is in the form of a disk 35 mm in diameter (1.38 in.) and 19 mm thick (0.75 in.).

Nitrogen analyses for value assignment were performed at NIST and cooperating laboratories using sample dissolution-Kjeldahl nitrogen determination, combustion-infrared detection, and vacuum-fusion, gas-volume measurement.

The certified value and uncertainty for nitrogen is reported as a mass fraction [1].

Certified Value of Nitrogen: 118.4 mg/kg ± 1.8 mg/kg

Certified Mass Fraction Value: 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 accounted for by NIST [2]. The certified value for nitrogen was determined by combining data from a primary method at NIST with two independent methods from cooperating laboratories. The uncertainty in the certified value is reported as ku_c , where k is the coverage factor for a 95 % confidence level and u_c is the combined standard uncertainty calculated according to the ISO/JCGM Guide [3]. The value of u_c is intended to represent, at the level of one standard deviation, the combined effect of all the uncertainties in the certified value. Since multiple independent analytical methods were used, the procedure of Schiller and Eberhardt was used to combine the data [4]. The certified value listed is metrologically traceable to the SI unit of mass, expressed as milligrams per kilogram.

Expiration of Certification: The certification of **SRM 1755** is valid indefinitely, within the measurement uncertainties 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 SRM over the period of its certification. If substantive technical changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of technical measurements for the certification of this SRM was performed by J.D. Fassett of the NIST Chemical Sciences Division.

Measurements for value assignment of SRM 1755 were performed by C.M. Beck II of the NIST Chemical Sciences Division.

Carlos A. Gonzalez, Chief Chemical Sciences Division

Robert L. Watters Jr., Director Office of Reference Materials

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Cooperative measurements were provided by C.L. Maul of Leco Corp., St. Joseph, MI, and L.W. Ollila and J.P. Flannigan of Luvak, Inc., Boylston, MA.

Statistical analysis of the certification data was provided by H-k. Liu of the NIST Statistical Engineering Division and homogeneity test data was provided by L.M. Gill of the NIST Statistical Engineering Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

Instructions for Use: Measurements should only be made in designated areas of the disk. The certified value is **NOT** valid within an area 1 cm (0.39 in.) in diameter located in the center and less than 5 mm (0.2 in) from the edge of the disk.

Alloy Preparation and Testing⁽¹⁾: The material for this SRM was provided by Bethlehem Steel Corp., Bethlehem, PA, and was prepared under the supervision of D.K. Vares of Bethlehem Steel Corp. Special attention was given to the material with regard to the desired nominal concentration of nitrogen (~100 mg/kg), homogeneity, and low titanium concentration; high concentrations of titanium in steel can produce an interference in the optical emission spectrometric determination of nitrogen. Homogeneity testing data were provided by the American Iron and Steel Institute (AISI) Technical Committee on Chemical Analysis under the supervision of D.K. Vares.

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/sp811/index.cfm (accessed Sep 2014).
- [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://www.nist.gov/srm/upload/SP260-136.PDF (accessed Sep 2014).
- [3] JCGM 100:2008; Evaluation of Measurement Data Guide to the Expression of Uncertainty in Measurement; (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 Sep 2014); 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://www.nist.gov/pml/pubs/index.cfm (accessed Sep 2014).
- [4] Schiller, S.B. and Eberhardt, K.R., *Combining Data from Independent Chemical Analysis Methods*, Spectrochimica Acta, 46B, pp. 1607-1613, (1991).

Certificate Revision History: 25 September 2014 (Extension of the certification period; editorial changes); 08 December 2000 (Original certification 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) 948-3730; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.

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⁽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.