National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 131g

Low Alloy Silicon Steel

(In Cooperation with the American Society for Testing and Materials)

This Standard Reference Material (SRM) is a low alloy, high silicon (nominally 3 %) steel intended primarily for checking and/or calibrating carbon/sulfur analyzers. One unit of SRM 131g consists of 150 g of chips, sized between 0.5 mm and 1.0 mm sieve openings (35 mesh and 18 mesh, respectively). The material designated SRM 131g is the last remaining portion of the same bulk material that was used to prepare SRM 131e and SRM 131f. Before bottling SRM 131g, the chips were reblended to reduce any inhomogeneities that may have developed during bulk storage.

Certified Values and Uncertainties: The certified values for carbon and sulfur, expressed as mass fractions [1], are listed in Table 1.

Table 1. SRM 131g Certified Values

Element	mg/kg
Sulfur	4.255 ± 0.052
Carbon	35.3 ± 2.2

The uncertainties listed in Table 1 are expanded uncertainties calculated according to the ISO/CIPM approach [2] at the 95 % level of confidence. The expanded uncertainties are calculated as $U = ku_c$ where u_c is the combined standard uncertainty and k is a coverage factor used to control the confidence level of the expanded uncertainty. For sulfur, u_c = 0.023 mg/kg, and represents at the level of one standard deviation, the combined effects of all known uncertainty associated with the mass spectrometry measurements, including the spike calibration and assay of standards. There were no differences in concentrations between SRM 131f and SRM 131g; therefore, all sulfur data were combined yielding a sample size of n = 23 and 16 degrees of freedom with a coverage factor of $k = 2.12$. For carbon, u_c = 1.03 mg/kg, and represents at the level of one standard deviation, the combined effects of uncertainty associated with the certification of SRM 131f and the intercomparison of SRM 131f and SRM 131g. Combining both the certification data for SRM 131f and the intercomparison data for SRM 131f and SRM 131g, gives 17.63 degrees of freedom for the combined standard uncertainty and a coverage factor of $k = 2.10$.

Expiration of Certification: The certification of SRM 131g is valid, within the measurement uncertainties specified until **28 February 2015**, provided the SRM is handled in accordance with the instructions given in this certificate (see Instructions for Use). This certification is nullified if the SRM is damaged, contaminated, or modified in any way other than its intended use.

Isotope dilution mass spectrometric sulfur measurements were performed by W.R. Kelly, J.L. Mann, and R.D. Vocke of the NIST Analytical Chemistry Division. The original carbon combustion-infrared detection determinations on SRM 131e were performed by T.A. Rush and T.W. Vetter of the NIST Analytical Chemistry Division. Additional carbon measurements were provided by the seven cooperating laboratories listed in Table 2. Subsequent combustion-infrared carbon measurements of both SRM 131f and SRM 131g were performed by D.A. Lawrenz of Leco Corporation, St. Joseph, MI.

The technical and support aspects involved in the preparation, certification, and issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by N.M. Trahey and C.R. Beauchamp.

Coordination of the technical measurements leading to the certification of SRM 131g was provided by J.D. Fassett of the NIST Analytical Chemistry Division.

Willie E. May, Chief
Analytical Chemistry Division

John Rumble, Jr., Acting Chief
Standard Reference Materials Program

Gaithersburg, MD 20899
Certificate Issue Date: 10 September 2001
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Statistical consultation was provided by W.F. Guthrie of the NIST Statistical Engineering Division.

INSTRUCTIONS FOR USE

Sampling: The unit should be mixed thoroughly by rotating the bottle before sampling. A minimum sample mass of 250 mg should be used for analytical determinations to be related to the sulfur value provided. For carbon measurements, the instrument manufacturer's recommendations for sampling should be consulted.

Alloy Preparation: The original material for SRM 131e, SRM 131f, and SRM 131g was obtained, under contract, from Armco Advanced Materials Corporation, Butler, PA, and forged, under contract, into six pieces by D.K. Associates, Buffalo, NY. After being tested for homogeneity, all six pieces were converted into chips at NIST and blended. Approximately half of the material was taken for characterization and certification, designated SRM 131e, and issued in 1991. To produce the subsequent issues of this SRM, the remaining bulk portion of chipped material was reblended and divided into two portions. One half, designated SRM 131f, was certified and issued in 1997. The remaining portion of material, designated SRM 131g, is described in this certificate.

PREPARATION, HOMOGENEITY, AND ANALYSIS

Sulfur measurements were made by isotope dilution thermal ionization mass spectrometry [3,4] on eight bottles of SRM 131g, selected by stratified random sampling, and four bottles of SRM 131f. The sulfur in samples of different masses was measured and the concentration determined by plotting the observed sulfur concentration versus mass of sample using a multivariate regression.

The carbon concentrations of SRM 131e and SRM 131f were directly compared by combustion-infrared detection during interlaboratory comparison study conducted in 1997. Triplicate carbon determinations were made, using a randomized run order, on six bottles of SRM 131e and six bottles of SRM 131f. Based on these data, the carbon value of SRM 131e was transferred directly to SRM 131f without modification. Quintuplicate carbon determinations were made in 2001 on one bottle of SRM 131f and two bottles of SRM 131g. Due to the excellent homogeneity and stability properties exhibited by this material, the carbon value for SRM 131f has been transferred to SRM 131g using current intercomparison data relating the carbon values of SRM 131f and SRM 131g.

Table 2. Cooperating Laboratories

C.K. Deak; C.K Deak Technical Services, Inc., Detroit, MI
C.C. Borland, R. Doebler, D. Gillum, and H. Vail; Armco Research and Technology, Middletown, OH
D. Hobson; Bethlehem Steel Corporation, Burns Harbor Plant, Chesterton, IN
L.W. Leonard and R. Hawkins; Inland Steel, East Chicago, IN
R. Hancock; Leco Corporation, St. Joseph, MI
J.M. Hlebek; LTV Steel Company, Indiana Harbor Works, East Chicago, IN
J.H. Morris and S. Forese; Lukens Steel Company, Coatesville, PA

REFERENCES

[1] Taylor, B.N., "Guide for the Use of the International System of Units (SI)," NIST Special Publication 811, 1995 Ed., (April 1995).

[2] *Guide to the Expression of Uncertainty in Measurement*, ISBN 92-67-10188-9, lst Ed. ISO, Geneva, Switzerland, (1993); see also Taylor, B.N. and 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/.

[3] Paulsen, P.J. and Kelly, W.R., "Determination of Sulfur as Arsenic Mono-sulfide Ion by Isotope Dilution Thermal Ionization Mass Spectrometry," Anal. Chem., **56**, pp. 708-713, (1984).

[4] Kelly, W.R. and Paulsen, P.J., "Precise and Accurate Determination of High Concentrations of Sulfur by Isotope Dilution Thermal Ionization Mass Spectrometry," Talanta, **31**, pp. 1063-1068, (1984).

Certificate Revision History: 10 September 2001 (This revision reflects an editorial change to the certificate); 20 May 2001 (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 at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet http://www.nist.gov/srm.