

Certificate of Analysis

Standard Reference Material[®] 36b

Chromium-Molybdenum Steel

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis. A unit of SRM 36b consists of a bottle containing approximately 150 g of chips.

Certified Values: Certified values for nine constituents in SRM 36b are provided in Table 1. All values are reported as mass fractions [1]. The uncertainty listed with the value is an expanded uncertainty, $U = ku_c$, based on a 95 % confidence level [2] and is calculated according to the method in the ISO Guide [3]. 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 [4]. A certified value is the present best estimate of the "true" value based on the results of analyses performed at NIST and collaborating laboratories. Test methods used to determine these elements are identified in the appendix and the accompanying key.

Reference Values: A reference value for carbon is provided in Table 2. 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 components of uncertainty [4]. The uncertainty listed with the value is an expanded uncertainty based on a 95 % confidence level [4] and is calculated according to the method in the ISO Guide [3].

Expiration of Certification: The certification of **SRM 36b** 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, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The original characterization of this material was performed in 1969 under the direction of O. Menis and J.I. Shultz of the National Bureau of Standards (NBS, now NIST).

Review and revision of value assignments was performed by J.R. Sieber and W.R. Kelly of the NIST Analytical Chemistry Division.

Statistical consultation for this SRM was provided by D.D. Leber 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: 08 September 2009 See Certificate Revision History on Last Page Analyses for certification were performed by the following: NBS: J.R. Baldwin, B.B. Bendigo, D.A. Becker, T.E. Gills, T.A. Rush, T.C. Rains, E.R. Deardorff, S.A. Wicks, T.J. Murphy, and W.R. Shields; Midvale-Heppenstall Company, Nicetown, Philadelphia, PA: W.L. MacBride; Universal-Cyclops Specialty Steel Division, Bridgeville, PA: R.C. Host, J. Kosek, and T. Hart; Weirton Steel Company, Weirton, WV: R.L. Zickefoose; and Latrobe Steel Company, Latrobe, PA: J.M. Henderson.

INSTRUCTIONS FOR USE

To relate analytical determinations to the certified values on this Certificate of Analysis, a minimum sample quantity of 200 mg is recommended. The material should be stored in its original container in a cool, dry location.

PREPARATION AND ANALYSIS¹

The material for this standard was provided by Lukens Steel Company, Coatesville, Pennsylvania. Analytical methods used for certification are provided in the appendix.

Constituent	Mass Fraction (%)	Expanded Uncertainty (Mass Fraction, %)	Coverage Factor, k
Cr	2.178	0.020	2.8
Cu	0.1792	0.0048	2.6
Mn	0.4041	0.0037	2.8
Mo	0.9960	0.0029	2.8
Ni	0.205	0.011	2.8
Р	0.0074	0.0021	2.8
S	0.01871	0.00085	2.8
Si	0.2580	0.0042	2.8
V	0.0043	0.0013	2.6

Table 1. Certified Values for SRM 36b Chromium-Molybdenum Steel

Table 2. Reference Value for SRM 36b Chromium-Molybdenum Steel

Constituent	Mass Fraction (%)	Expanded Uncertainty (Mass Fraction, %)	Coverage Factor, k
С	0.1143	0.0024	2.8

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.

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 <u>http://physics.nist.gov/Pubs/</u>.
- [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, U.S. Government Printing Office, Washington, DC, p. 16 (2000); available at http://www.cstl.nist.gov/nist839/NIST_special_publications.htm.
- [3] JCGM 100:2008; Guide to the Expression of Uncertainty in Measurement; (ISO GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology: BIPM, Sevres Cedex, France (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf; 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.physics.nist.gov/Pubs/contents.html.
- [4] Hahn, G.J.; Meeker, W.Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc., New York (1991).

Certificate Revision History: 08 September 2009 (This revision reports revised assignments and values for all constituents based on reevaluation of the original analytical results and updates the entire certificate to current NIST standards); 18 July 1969 (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) 975-2200; fax (301) 926-4751, email srminfo@nist.gov; or via the Internet at <u>http://www.nist.gov/srm</u>.

Appendix. Analytical Methods

Element	Methods*
С	20
Cr	7, 17
Cu	5, 6, 11, 19
Mn	1, 9, 18
Мо	15, 21
Ni	12, 16, 21
Р	2, 10, 14
S	3
Si	4
V	1, 8, 13

*Key to Methods:

- 1. Neutron activation analysis
- 2. Molybdenum-blue photometric method. See J. Res. Natl. Bur. Stand. 26, No. 5, 405 (1941) RP 1386
- 3. 1 g sample burned in oxygen at 1450 °C and sulfur dioxide absorbed in starch-iodide solution. Iodine liberated from iodide by titration during the combustion with standard KIO₃ solution.
- 4. Perchloric acid double dehydration.
- 5. Isotope dilution method
- 6. Atomic absorption method
- 7. Persulfate oxidation-potentiometric titration with ferrous ammonium sulfate
- 8. Benzoylphenylhydroxylamine photometric method
- 9. Bismuthate oxidation-potentiometric titration with HgNO₃
- 10. Alkali molybdate method
- 11. Neocuproine photometric method
- 12. Dimethylglyoxime precipitate titrated with cyanide
- 13. Nitric acid oxidation-potentiometric titration with standard ferrous ammonium sulfate solution
- 14. Color complex extracted with isobutyl alcohol
- 15. Alpha benzoinoxime-MoO₃ method
- 16. Electrolytic method
- 17. Persulfate oxidation- $Fe(NH_4)_2(SO_4)_2 KMnO_4$ titration
- 18. Persulfate photometric method after removal of chromium as CrO₂Cl₂
- 19. Diethyldithiocarbamate photometric method
- 20. Combustion-gravimetry
- 21. Photometric