National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 50c

Tungsten, Chromium, Vanadium Steel

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis. A unit of SRM 50c consists of a bottle containing approximately 150 g of chips.

Certified Values: Certified values for 13 constituents in SRM 50c 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 Value: A reference value for carbon is given in Table 2. A reference value is a non-certified values that is the present best estimate of the true value; however, the value does not meet the NIST criteria for certification and is provided with associated uncertainty 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]. Test method used to determine carbon is identified in the appendix and the accompanying key.

Information Value: An information value is given for cobalt 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. The value is intended to provide additional information on the matrix.

Expiration of Certification: The certification of **SRM 50c** 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 before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The overall direction and coordination leading to the certification of this SRM was performed by J.I. Shultz of the National Bureau of Standards (NBS, now NIST).

Review and revision of value assignments were 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.

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Analyses for certification were performed by the following: NBS: W.R. Kelly, E.J. Maienthal, R.E. McIntyre, P.J. Paulsen, J.I. Shultz , and L.J. Tregoning; Allegheny Ludlum Steel Corporation, Dunkirk, NY: A.B. Cargill; The Cleveland Twist Drill Company, Cleveland, OH: W.L. Emerson; H. K. Porter Company, Inc., Henry Disston Division, Philadelphia, PA.: E.F. Nunemacher and A.R. Uhl; Crucible Steel Company of America, Sanderson-Halcomb Works, Syracuse, NY: O.L. Van Valkenburgh and S.M. Dibble; Borg-Warner Corporation, Atkins Saw Division, Indianapolis, IN: H.A. Burkhardt; Atlas Steels Limited, Welland, Canada: E. Jackman, J. Sernasie, and G. Laidlaw; Bethlehem Steel Company, Inc., Bethlehem, PA: W.F. Zollinger; Electro Metallurgical Company, Niagara Works, Niagara Falls, NY: J.J. Furey; Carpenter Steel Company, Reading, PA: M.J. Noll, A.L. Sloan, and J.O. Strauss.

INSTRUCTIONS FOR USE

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

Table 1. Certified Values for SRM 50c

Constituent	Mass Fraction (%)	Expanded Uncertainty (%)	Coverage Factor, *k*
As	0.0225	0.0050	2.6
Cr	4.128	0.011	2.3
Cu	0.0792	0.0026	2.3
Mn	0.3417	0.0044	2.3
Mo	0.0821	0.0056	2.3
N	0.0117	0.0014	4.3
Ni	0.0686	0.0032	2.3
P	0.0222	0.0010	2.2
S	0.006 367	0.000 094	2.6
Si	0.3102	0.0089	2.3
Sn	0.0183	0.0028	2.4
V	1.158	0.013	2.3
W	18.445	0.050	2.3

Table 2. Reference Value for SRM 50c

Constituent	Mass Fraction (%)	Expanded Uncertainty (%)	Coverage Factor, *k*
C	0.7193	0.0034	2.3

Table 3. Information Value for SRM 50c

Constituent	Mass Fraction (%)
Co	0.035

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 Feb 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 Feb 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 Feb 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. Goverment Printing Office: Washington, DC (1994); available at http://physics.nist.gov/Pubs/ (accessed Feb 2011).

[4] Hahn, G.J.; Meeker, W.Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc.: New York (1991).

[5] J. Res. Natl. Bur. Stand.; Vol. 26, p. 405 (1941) RP1386.

[6] Paulsen, P.J.; Kelly, W.R.; *Determination of Sulfur as Arsenic Monosulfide ion by Isotope Dilution Thermal Ionization Mass Spectrometry*; Anal. Chem., Vol. 56, pp. 708–713 (1984).

[7] J. Res. Natl. Bur. Stand.; Vol. 47, p. 380 (1951) RP2265.

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

[9] J. Res. Natl. Bur. Stand.; Vol. 24, p. 7 (1940) RP1267.

[10] J. Res. Natl. Bur. Stand.; Vol. 43, p. 201 (1949) RP2021.

Certificate Revision History: 17 February 2011 (This revision includes editorial revisions); 23 September 2009 (This revision reports revised assignments and values for all constituents based on re-evaluation of the original analytical results and updates the entire certificate to current NIST standards); 06 September 2001 (editorial revision); 25 June 1957 (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.

APPENDIX

Analytical Methods

Element	Methods[(a)]
As	30, 31, 32, 33
C	Direct Combustion
Cr	6, 20
Cu	14, 15, 16, 17
Mn	1, 2, 3, 4, 5, 6, 7
Mo	23
N	34
Ni	18, 19
P	6, 8, 9
S	10
Si	11, 12, 13
Sn	6, 26, 28, 29
V	6, 21, 22
W	24, 25, 27

[(a)]Key to Methods:

1. Potentiometric titration
2. Bismuthate - $FeSO_4 – KMnO_4$
3. Chromium separated with ZnO
4. Periodate photometric method
5. Potentiometric titration with $HgNO_3$
6. Titrating solution standardized by the use of a standard steel
7. Chromium volatilized as CrO_2Cl_2
8. Weighed as ammonium phosphomolybdate
9. Molybdenum-blue photometric method [5]
10. Isotope dilution thermal ionization mass spectrometry [6]
11. Double dehydration with intervening filtration
12. Nitric-hydrochloric acid digestion
13. H_2SO_4 Dehydration
14. Diethyldithiocarbamate photometric method [7]
15. Neo-cuproine colorimetric method
16. Copper precipitated with Na_2S2O_3
17. Finished by electrolysis
18. Dimethylglyoxime precipitate titrated with cyanide
19. Dimethylglyoxime photometric method
20. Persulfate oxidation, potentiometric titration with $FeSO_4–(NH_4)_2S_2O_8$
21. $FeSO_4– (NH_4)_2S_2O_8 – KMnO_4$ method
22. Vanadium oxidized with $KMnO_4$
23. $H_2S – MoS_3 – MoO_3$
24. Tungsten precipitation by acid digestion and cinchonine. Ignited WO_3 corrected for Si, Fe, Cr, V, and Mo
25. Hydroquinone photometric method
26. Sulfide-iodine method [8]
27. Major portion of tungsten precipitated by hydrolysis and cinchonine in $HCl – HNO_3 – HClO_4$ solution. Alphabenzoinoxime added. WO_3 corrected for Mo, V, NaOH, and NH_4OH insolubles.
28. Polargraphic method
29. $H_2S – SnS_2 – SnO_2$
30. Distillation – molybdenum blue photometric method [9].
31. Distillation – $As_2S_3 – Ag_3AsO_4$ – KSCN titration method
32. Distillation – titration with standard $KBrO_3$
33. Distillation – titration with standard $KMnO_4$
34. Sulfuric acid digestion for 3 h of a 1 g sample [10].