National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 32e

Nickel-Chromium Steel
(SAE 3140)

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis. A unit of SRM 32e consists of a bottle containing approximately 150 grams of chips.

Certified Values: Certified values for nine constituents in SRM 32e 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 vanadium is given 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].

Information Values: Information values are provided for two constituents 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. They are intended to provide additional information on the matrix.

Expiration of Certification: The certification of **SRM 32e** 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 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 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.

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Certificate Issue Date: 24 September 2009
See Certificate Revision History on Page 3

Robert L. Watters, Jr., Chief
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Analyses for certification were performed by the following: NBS: R.E. McIntyre, E.J. Maienthal, J.R. Spann, and L.J. Tregoning; Copperweld Steel Company, Warren, OH: R.F. Lab and D.R. Burrier; Midvale-Heppenstall Company, Nicetown, PA: J.A. Wiley; Great Lakes Steel Corporation, Ecorse, Detroit, MI: R.D. O'Mara; United Engineering and Foundry Company, Vandergrift, PA: C.H. Cramer; General Electric Corporation, Transformer Laboratories, Pittsfield, MA: R.R. Ralston, J.W. Fulton, A.M. Hunt, J.P. Broyles, and R.J. Londergan; United States Steel Corporation, Lorain Works, Lorain, OH: A.J. Kielar; and United States Steel Corporation, Gary Works, Gary IN: O.W. Baldwin.

INSTRUCTIONS FOR USE

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

PREPARATION AND ANALYSIS[1]

The material for this standard was furnished by the Copperweld Steel Company. Analytical methods used for certification are provided in the appendix.

Table 1. Certified Values for SRM 32e Nickel-Chromium Steel

Constituent	Mass Fraction (%)	Expanded Uncertainty (Mass Fraction, %)	Coverage Factor, *k*
C	0.4086	0.0036	2.4
Cr	0.6775	0.0044	2.4
Cu	0.1266	0.0029	2.4
Mn	0.7983	0.0049	2.4
Mo	0.0228	0.0020	2.4
Ni	1.1938	0.0099	2.4
P	0.00888	0.00091	2.4
S	0.0210	0.0012	2.4
Si	0.2775	0.0031	2.4

Table 2. Reference Values SRM 32e Nickel-Chromium Steel

Constituent	Mass Fraction (%)	Expanded Uncertainty (Mass Fraction, %)	Coverage Factor, *k*
V	0.00225	0.00087	2.4

Table 3. Information Values for SRM 32e Nickel-Chromium Steel

Constituent	Mass Fraction (%)
N	0.009
Sn	0.011

[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 http://physics.nist.gov/Pubs/.

[2] May, W. E.; Parris, R. M.; B*eck II, C. M.; Fassett, J. D.; Greenberg, R. R.; Guenthe*r, *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).

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

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

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

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

Certificate Revision History: 24 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); 05 April 1957 (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 http://www.nist.gov/srm.

Appendix. Analytical Methods

Element	Additional Method(s)*	Principle Method(s)
C	25	Direct combustion
Cr	8, 18, 23, 27, 31	$FeSO_4$ – $KMnO_4$ titration
Cu	7, 13, 16, 22, 30	H_2S – CuS – CuO
Mn	3, 12, 15, 29	Bismuthate ($FeSO_4$ – $KMnO_4$) Persulfate – arsenite
Mo	37	Colorimetric
N	10	Distillation – titration
Ni	17	Weighed as nickel dimethylglyoxime
P	1, 4, 12, 20	Gravimetric (weighed as $Mg_2P_2O_7$ after removal of arsenic) Alkali – molybdate
S	5, 9, 12, 21, 24, 26, 35	Gravimetric (direct oxidation and precipitation after reduction of iron) Combustion iodate titration Evolution with HCl (1 – 1) ZnS – iodine (theoretical sulfur titer)
Si	6, 7, 33	Perchloric acid dehydration
Sn	11	
V	2, 14, 19, 24, 26, 28, 32, 34, 36	

*Key to Methods:

1. Precipitated at 40 °C, washed with a 1 % solution of KNO_3 and titrated with alkali standardized by the use of acid potassium phthalate and the ratio 23 NaOH:P.
2. Value obtained by standardizing the titrating solution by means of sodium oxalate through $KMnO_4$ and $Na_2S_2O_3$ and use of the ratio 21:1S.
3. Potentiometric titration
4. Molybdenum-blue photometric method [5].
5. 1 g sample burned in O_2 at 1425 °C, and SO_2 absorbed in starch-iodide solution. Iodine liberated from iodide by titration, during the combustion, with standard KIO_3 solution. Titer based on 93 % of the theoretical factor.
6. Double dehydration with intervening filtration.
7. Diethyldithiocarbamate photometric method [6].
8. Chromium separated from the bulk of the iron in a 5 g sample by hydrolytic precipitation with $NaHCO_3$, oxidized with persulfate, and titrated potentiometrically with $FeSO_4$-$(NH_4)_2S_2O_8$.
9. Vanadium separated as in method (8), oxidized with HNO_3 and titrated potentiometrically with $FeSO_4$-$(NH_4)_2S_2O_3$.
10. Sulfuric acid digestion for 4 h of a 1 g sample [7].
11. Sulfide-iodine method [8].
12. Titrating solution standardized with standard steel.
13. KI – $Na_2S_2O_3$ titration.
14. Vanadium coprecipitated with phosphomolybdate reduced with H_2O_2 in fuming H_2SO_4, and titrated with $KMnO_4$.
15. Potentiometric titration with $HgNO_3$.
16. Copper precipitated with $Na_2S_2O_3$, finished by electrolysis.
17. Dimethylglyoxime precipitate titrated with KCN.
18. Persulfate oxidation, potentiometric titration with $FeSO_4$-$(NH_4)_2S_2O_3$.
19. Nitric acid oxidation, potentiometric titration with $FeSO_4$-$(NH_4)_2S_2O_3$.
20. Molybdenum-blue photometric method. Colored complex extracted into $(CH_3)_3CH_3OH$ and measured at 730 nm.
21. Absorbed in ammoniacal $CdCl_2$.
22. Finished by electrolysis.
23. Perchloric acid oxidation, titration with $FeSO_4$-$KMnO_4$ using *o*-phenathroline indicator.
24. Vanadium separated with cupferron and determined by $FeSO_4$ - $(NH_4)_2S_2O_3$ – $KMnO_4$ method.
25. Differential gasometric method.
26. Solution in diluted HCl (2+1).
27. Persulfate oxidation.
28. Bicarbonate separation. Chromium volatilized with HCl-$HClO_4$. Vanadium reduced with $FeSO_4$ and titrated in buffered solution with $KMnO_4$.
29. Periodate photometric method.
30. Neocuproine photometric method.
31. Perchloric acid oxidation, titration with $FeSO_4$ – $Ce(SO_4)_2$.
32. $NaHCO_3$ hydrolysis followed by Hg cathode. Vanadium determined by the phosphotungstovanadate photometric method.
33. Sulfuric acid dehydration.
34. Vanadium separated as in method (32), and titrated with $KMnO_4$.
35. Sulfur gases absorbed in neutral H_2O_2, and H_2SO_4 titrated with standard NaOH using bromcresol green indicator.
36. As in method (14), except V reduced with $FeSO_4$ and titrated with $KMnO_4$.
37. H_2S - MoS_4 - MoO_3.