



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 32e

Carbon Low Alloy Steel (SAE 3140) (chip form)

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 g of chips.

Certified Values: The measurand is the total elemental content for each constituent in SRM 32e. Certified values are provided in Table 1 and are reported as mass fractions [1]. The uncertainty listed with the value is an expanded uncertainty, $U = k_{uc}$, based on a 95 % confidence level [2] and is calculated according to the method in the ISO/JCGM 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 the constituents are identified in the appendix and the accompanying key. Metrological traceability is to the derived SI unit for mass fraction (expressed as a percent).

Reference Value: The measurand is the total content of vanadium provided in Table 2 as determined by the indicated methods. 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/JCGM Guide [3]. Metrological traceability is to the derived SI unit for mass fraction (expressed as a percent).

Information Values: Information values are provided for additional 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. Information values cannot be used to establish metrological traceability.

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 or register online) will facilitate notification.

Coordination of technical measurements for 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 Chemical Sciences 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 Office of Reference Materials.

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Gaithersburg, MD 20899
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Steven J. Choquette, Acting Director
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Analyses for certification were performed by the following: R.E. McIntyre, E.J. Maienthal, J.R. Spann, and L.J. Tregoning (NBS); R.F. Lab and D.R. Burrier (Copperweld Steel Company, Warren, OH); J.A. Wiley (Midvale-Heppenstall Company, Nicetown, PA); R.D. O'Mara (Great Lakes Steel Corporation, Ecorse, Detroit, MI); C.H. Cramer (United Engineering and Foundry Company, Vandergrift, PA); R.R. Ralston, J.W. Fulton, A.M. Hunt, J.P. Broyles, and R.J. Londergan (General Electric Corporation, Transformer Laboratories, Pittsfield, MA); A.J. Kielar (United States Steel Corporation, Lorain Works, Lorain, OH); and O.W. Baldwin (United States Steel Corporation, Gary Works, Gary, IN).

INSTRUCTIONS FOR USE

To relate analytical determinations to the certified values on this certificate, 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 Carbon Low Alloy Steel (SAE 3140)

| Constituent | Mass Fraction (%) | Expanded Uncertainty (%) | Coverage Factor (<i>k</i>) |
|-----------------|----------------------|-----------------------------|---------------------------------|
| Carbon (C) | 0.4086 | 0.0036 | 2.4 |
| Chromium (Cr) | 0.6775 | 0.0044 | 2.4 |
| Copper (Cu) | 0.1266 | 0.0029 | 2.4 |
| Manganese (Mn) | 0.7983 | 0.0049 | 2.4 |
| Molybdenum (Mo) | 0.0228 | 0.0020 | 2.4 |
| Nickel (Ni) | 1.1938 | 0.0099 | 2.4 |
| Phosphorus (P) | 0.00888 | 0.00091 | 2.4 |
| Sulfur (S) | 0.0210 | 0.0012 | 2.4 |
| Silicon (Si) | 0.2775 | 0.0031 | 2.4 |

Table 2. Reference Value for SRM 32e Carbon Low Alloy Steel (SAE 3140)

| Constituent | Mass Fraction (%) | Expanded Uncertainty (%) | Coverage Factor (<i>k</i>) |
|--------------|----------------------|-----------------------------|---------------------------------|
| Vanadium (V) | 0.00225 | 0.00087 | 2.4 |

Table 3. Information Values for SRM 32e Carbon Low Alloy Steel (SAE 3140)

| Constituent | Mass Fraction (%) |
|--------------|----------------------|
| Nitrogen (N) | 0.009 |
| Tin (Sn) | 0.011 |

⁽¹⁾ Certain commercial equipment, instrumentation, 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 www.nist.gov/pml/pubs/index.cfm/ (accessed Oct 2016).
- [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: Washington, DC (2000); available at <http://www.nist.gov/srm/upload/SP260-136.PDF> (accessed Oct 2016).
- [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/utls/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Oct 2016); 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 Oct 2016).
- [4] Hahn, G.J.; Meeker, W.Q.; *Statistical Intervals: A Guide for Practitioners*; John Wiley & Sons, Inc.: New York (1991).
- [5] (1941) RP1386.
- [6] J. Res. Natl. Bur. Stand.; Vol. 47, 380 (1951) RP2265.
- [7] J. Res. Natl. Bur. Stand.; Vol. 43, 201 (1949) RP2021.
- [8] J. Res. Natl. Bur. Stand.; Vol. 8, 309 (1932) RP415.

Certificate Revision History: 20 October 2016 (Element name for N corrected in Table 3; editorial changes) 19 August 2016 (Editorial changes); 07 July 2016 (Updated title; editorial changes); 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 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 at <http://www.nist.gov/srm>.

Appendix. Analytical Methods

| Constituent | 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 ₂ S – 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 ₂ P ₂ O 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 KNO₃ 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 KMnO₄ and Na₂S₂O₃ and use of the ratio 21:1S.
3. Potentiometric titration
4. Molybdenum-blue photometric method [5].
5. 1 g sample burned in O₂ at 1425 °C, and SO₂ absorbed in starch-iodide solution. Iodine liberated from iodide by titration, during the combustion, with standard KIO₃ solution. Titer based on 93 % of the theoretical factor.
6. Double dehydration with intervening filtration.
7. Diethylthiocarbamate photometric method [6].
8. Chromium separated from the bulk of the iron in a 5 g sample by hydrolytic precipitation with NaHCO₃, oxidized with persulfate, and titrated potentiometrically with FeSO₄-(NH₄)₂S₂O₈.
9. Vanadium separated as in method (8), oxidized with HNO₃ and titrated potentiometrically with FeSO₄-(NH₄)₂S₂O₃.
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 – Na₂S₂O₃ titration.
14. Vanadium coprecipitated with phosphomolybdate reduced with H₂O₂ in fuming H₂SO₄, and titrated with KMnO₄.
15. Potentiometric titration with HgNO₃.
16. Copper precipitated with Na₂S₂O₃, finished by electrolysis.
17. Dimethylglyoxime precipitate titrated with KCN.
18. Persulfate oxidation, potentiometric titration with FeSO₄-(NH₄)₂S₂O₃.
19. Nitric acid oxidation, potentiometric titration with FeSO₄-(NH₄)₂S₂O₃.
20. Molybdenum-blue photometric method. Colored complex extracted into (CH₃)₃CH₃OH and measured at 730 nm.
21. Absorbed in ammoniacal CdCl₂.
22. Finished by electrolysis.
23. Perchloric acid oxidation, titration with FeSO₄-KMnO₄ using *o*-phenathroline indicator.
24. Vanadium separated with cupferron and determined by FeSO₄ - (NH₄)₂S₂O₃ – KMnO₄ method.
25. Differential gasometric method.
26. Solution in diluted HCl (2+1).
27. Persulfate oxidation.
28. Bicarbonate separation. Chromium volatilized with HCl-HClO₄. Vanadium reduced with FeSO₄ and titrated in buffered solution with KMnO₄.
29. Periodate photometric method.
30. Neocuproine photometric method.
31. Perchloric acid oxidation, titration with FeSO₄ – Ce(SO₄)₂.
32. NaHCO₃ 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 KMnO₄.
35. Sulfur gases absorbed in neutral H₂O₂, and H₂SO₄ titrated with standard NaOH using bromcresol green indicator.
36. As in method (14), except V reduced with FeSO₄ and titrated with KMnO₄.
37. H₂S - MoS₄ - MoO₃.