National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 121d

Stainless Steel (Cr 17-Ni 11-Ti 0.3)
(AISI 321)

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis. A unit of SRM 121d consists of a bottle containing approximately 150 g of chips. This material is also available in disk form as SRM 1171 for applications in optical emission spectrometry and X-ray fluorescence spectrometry.

Certified Values: Certified values for eight constituents in SRM 121d 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 one constituent 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].

Information Values: Information values are provided for three 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 121d** 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 1971 under the direction of O. Menis and J.I. Shultz of the National Bureau of Standards (NBS, now NIST). Homogeneity testing was performed by S.D. Rasberry, M. McKay, and B.F. Scribner of NBS.

Review and revision of value assignments was performed by W.R. Kelly and J.R. Sieber 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

Gaithersburg, MD 20899
Certificate Issue Date: 08 September 2009
See Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
Measurement Services Division

Analyses for value assignment were performed by the following: NBS: R.K. Bell, T.A. Rush, T.C. Rains, and S.A. Wicks; Armco Steel Corporation, Baltimore, MD: R.L. LeRoy and L.V. Beauchamp; and Carpenter Technology Corporation, Research and Development Center, Reading, PA: J.O. Strauss.

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. Specimens may be used directly from the bottle without pre-treatment. The material should be stored in its original container in a cool, dry location.

PREPARATION AND ANALYSIS[1]

The material for this standard was prepared at the Duquesne Works, U.S. Steel Corporation, Pittsburgh, PA. Homogeneity testing was performed using X-ray fluorescence spectrometry. Certification analyses were performed using the methods provided in the appendix.

Table 1. Certified Values for SRM 121d Stainless Steel (Cr 17-Ni 11-Ti 0.3)

Constituent	Mass Fraction (%)	Expanded Uncertainty (Mass Fraction, %)	Coverage Factor, *k*
C	0.067	0.010	12.7
Cr	17.50	0.15	3.2
Cu	0.1205	0.0057	12.7
Mn	1.81	0.16	12.7
Mo	0.167	0.040	12.7
Ni	11.18	0.21	12.7
Si	0.536	0.011	12.7
Ti	0.346	0.057	12.7

Table 2. Reference Values for SRM 121d Stainless Steel (Cr 17-Ni 11-Ti 0.3)

Constituent	Mass Fraction (%)	Expanded Uncertainty (Mass Fraction %)	Coverage Factor, *k*
Co	0.097	0.079	12.7

Table 3. Information Values for SRM 121d Stainless Steel (Cr 17-Ni 11-Ti 0.3)

Constituent	Mass Fraction (%)
P	0.019
S	0.013
W	0.012

[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).

Certificate Revision History: 08 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); Revision 31 August 1981; 07 July 1971 (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	Methods*
C	4
Co	1, 11
Cr	1, 14, 15
Cu	1
Mn	3, 12
Mo	2, 13
Ni	6
P	2
S	5
Si	7, 8
Ti	10
W	9

*Key to Methods:

1. Atomic absorption spectroscopy
2. Colorimetric – direct
3. Colorimetric – oxidation with ammonium persulfate with $AgNO_3$
4. Combustion – gas chromatography
5. Combustion – iodometric titration
6. Gravimetric – dimethylglyoxime, NaCN titration
7. Gravimetric – perchloric acid – double dehydration
8. Gravimetric – perchloric acid – single dehydration
9. Photometric – a-benzoinoxime with Mo, fluoride ion exchange, dithio extraction, spectrophotometric
10. Photometric – spectrophotometric diantipyrrylmethane
11. Photometric – spectrophotometric nitroso R
12. Photometric – spectrophotometric periodate
13. Photometric – spectrophotometric thiocyanate
14. Tritrimetric – persulfate oxidation and potentiometric titration with $FeSO_4$-$(NH_4)_2S_2O_8$
15. Volumetric – oxidation with ammonium persulfate, reduced with $FeSO_4$-$(NH_4)_2S_2O_8$, titrated with $KMnO_4$