National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 25d

Manganese Ore

(In cooperation with ASTM International)

This Standard Reference Material (SRM) is intended primarily for use in validation of chemical and instrumental methods of analysis. A unit of SRM 25d consists of a bottle containing approximately 60 g of powder of particle size less than 74 μm (No. 200 sieve).

Certified Values: Certified values for three constituents in SRM 25d are listed 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: Reference values for five constituents are listed 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 for three constituents are listed 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 25d** 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 1984 under the direction of J.I. Shultz of the National Bureau of Standards (NBS, now NIST). Homogeneity testing was performed by G. Marinenko at NBS.

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

Gaithersburg, MD 20899
Certificate Issue Date: 26 May 2010
See Certificate Revision History on Last Page

Robert L. Watters, Jr., Chief
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Analyses for certification were performed by the following: NBS: G. Marinenko, Y.C. Wu, and R.L. Watters, Jr.; Andrew McGreath & Sons, Inc., Harrisburg, PA; F.A. Pennington, Jr., L.W. Richards, F. Hagenberger, C. Jacoby, and G. Bruno; Elkem Metals Company, Marietta, OH, H.H. Hall; and Ledoux & Company, Teaneck, NJ, S. Kallman and C.L. Maul.

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. A portion of the material to be analyzed must be dried for 2 h at 120 °C prior to weighing. The material should be stored in its original container in a cool, dry location.

PREPARATION, TESTING, ANALYSIS [1]

SRM 25d was produced in cooperation with ASTM International Committee E01 Analytical Chemistry of Metals, Ores and Related Materials. The material for the preparation of this SRM was prepared by Elkem Metals Company, Marietta, OH.

Homogeneity testing was based on the determination of total manganese by the sodium bismuthate oxidation procedure. The material heterogeneity is less than can be detected by the analytical method (≤ 0.2 % relative). Analytical methods used for certification are provided in the appendix.

Table 1. Certified Values for SRM 25d Manganese Ore

Constituent	Mass Fraction (%)	Expanded Uncertainty (Mass Fraction, %)	Coverage Factor, *k*
Mn	51.78	0.17	3.2
P_2O_5	0.251	0.015	3.2
SiO_2	2.54	0.14	3.2

Table 2. Reference Values for SRM 25d Manganese Ore

Constituent	Mass Fraction (%)	Expanded Uncertainty (Mass Fraction, %)	Coverage Factor, *k*
Al_2O_3	5.33	0.21	4.3
Fe_2O_3	3.91	0.18	4.3
K_2O	0.928	0.041	4.3
TiO_2	0.136	0.030	4.3
Available Oxygen	14.283	0.081	3.2

Table 3. Information Values for SRM 25d Manganese Ore

Constituent	Mass Fraction (%)
BaO	0.21
CaO	0.052
Moisture	1

[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://ts.nist.gov/WeightsAndMeasures/Metric/mpo_pubs.cfm (accessed May 2010).

[2] May, W. E.; Parris, R. M.; B*eck 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; *Evaluation of Measurement Data — Guide to the Expression of Uncertainty in Measurement* (ISO GUM 1995 with Minor Corrections); Joint Committee for Guides in Metrology (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed May 2010); 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/physlab/pubs/index.cfm (accessed May 2010).

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

Certificate Revision History: 26 May 2010 (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); 10 February 1984 (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*
Al_2O_3	5
BaO	9
CaO	9
Fe_2O_3	5
K_2O	5, 6
Mn	1
P_2O_5	2, 4
SiO_2	2, 3
TiO_2	7
Available Oxygen	8

*Key to Methods:

1. Sodium bismuthate oxidation
2. Inductively coupled plasma optical emission spectrometry
3. Acid dehydration and gravimetry
4. Photometric method
5. Atomic absorption spectrophotometry
6. Flame emission spectrophotometry
7. Colorimetry
8. $Fe(NH_4)_2(SO_4)_2$ reduction – $KMnO_4$ titration
9. Direct current plasma optical emission spectrometry