National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2451

Fine Carbon (Activated) – From Cyanide Ore Leaching

This Standard Reference Material (SRM) is intended for use in the evaluation of chemical methods of analysis and in calibration of instrumental methods of analysis. A unit of SRM 2451 consists of a bottle containing 100 g of fine-powder carbon (activated) obtained after use in the leaching of ore with cyanide solution.

Certified Value: The certified mercury content, provided in Table 1, is based on analyses by cold-vapor isotope dilution inductively coupled plasma mass spectrometry (CV-ID-ICP-MS) [1,2]. 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 [3]. The expanded uncertainty for the certified value for mercury is calculated as a 95 % confidence interval where $U = ku_c$. The quantity u_c is intended to represent, at the level of one standard deviation, the combined standard uncertainty calculated according to the ISO and NIST Guides [4]. The coverage factor, $k = 1.97$, corresponds to a t factor for approximately 360 degrees of freedom.

Table 1. Certified Value [(a)] (Mass Fraction)

Mercury (Hg)	688 mg/kg ± 28 mg/kg

[(a)] Dry-mass basis (see "Instructions for Use")

Reference Value: The reference gold content, provided in Table 2, is based on analyses by inductively coupled plasma mass spectrometry (ICP-MS). A reference value is a non-certified value that is the present best estimate of the true value [3]. However, the value does not meet the NIST criteria for certification and is provided with an associated uncertaintiy that may not include all sources of uncertainty. The expanded uncertainty for the reference value for gold is calculated as a 95 % confidence interval where $U = ku_c$. The quantity u_c is intended to represent, at the level of one standard deviation, the combined standard uncertainty calculated according to the ISO and NIST Guides [4]. The coverage factor, $k = 1.98$, corresponds to a t factor for approximately 110 degrees of freedom.

Table 2. Reference Value [(a)] (Mass Fraction)

Gold (Au)	28.0 mg/kg ± 1.5 mg/kg

[(a)] Dry-mass basis (see "Instructions for Use")

Information Value: An information value for cyanide is provided in Table 3. The cyanide determination was performed by a commercial laboratory using EPA method SW-846 9012a. Insufficient information is available to assess adequately its uncertainty. It is given only to provide additional information on the matrix.

Table 3. Information Value

Cyanide (CN^-)	96 mg/kg

Expiration of Certification: The certification of SRM 2451 is valid, within the measurement uncertainty specified, until **31 December 2014**, provided the SRM is handled in accordance with instructions given in this certificate (see "Instructions for Use"). The certification is nullified if the SRM is damaged, contaminated, or otherwise modified.

Coordination of the technical measurements for certification of SRM 2451 was accomplished under the direction of W.R. Kelly of the NIST Analytical Chemistry Division.

Stephen A. Wise, Chief
Analytical Chemistry Division

Gaithersburg, MD 20899
Certificate Issue Date: 15 January 2009
See Certificate Revision History on Last Page

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

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.

Analytical measurements for certification of this SRM were performed by W.R. Kelly, S.E. Long, J.L. Mann, A.F. Marlow, and J.R. Sieber of the NIST Analytical Chemistry Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Measurement Services Division.

INSTRUCTIONS FOR USE

The material should be stored in its original, tightly sealed container until use. The recommended minimum test portion is 100 mg, which was the quantity used for determinations of Hg and Au at NIST. The material should be dried at a temperature between 105 °C and 110 °C in an atmosphere of dry, high-purity N_2 gas. The typical mass loss on drying obtained at NIST was 15.4 %

PLANNING, PREPARATION, TESTING, AND ANALYSIS[1]

The material for SRM 2451 was obtained from Newmont Mining Corp., Denver, CO. Typical material such as this begins as relatively pure carbon obtained by the destructive distillation of wood, nutshells, animal bones, or other carbonaceous material. It is activated by heating to 800 °C to 900 °C with steam or carbon dioxide. Activation results in a porous structure with high internal surface area that imparts a high adsorptivity for many gases, vapors, colloidal solids, and metals. The material for SRM 2451 was used in conjunction with cyanide solution in the extraction of metals from gold ore and contains absorbed cyanide and metal compounds produced in the gold leaching process.

The material was blended and bottled by the NIST Measurement Services Division. After bottling, homogeneity testing was performed by A.F. Marlow of the NIST Analytical Chemistry Division using X-ray fluorescence spectrometry. The homogeneity test results showed no additional variance beyond that represented by the variance of individual results obtained for the material using the ICP-MS test methods described above.

REFERENCES

[1] Christopher, S.J.; Long, S.E.; Rearick, M.S.; Fassett, J.D.; *Development of Isotope Dilution Cold Vapor Inductively Coupled Plasma Mass Spectrometry and Its Application to the Certification of Mercury in NIST Standard Reference Materials*; Anal. Chem., Vol. 73, pp. 2190–2199 (2001).

[2] Long, S.E.; Kelly, W.R.; *Determination of Mercury in Coal by Isotope Dilution Cold-Vapor Generation Inductively Coupled Plasma Mass Spectrometry*, Anal. Chem., Vol. 74, pp. 1477–1483(2002).

[3] May, W.E.; Parris, R.M.; Beck 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, p.16; U.S. Government Printing Office: Washington, DC (2000); available at http://ts.nist.gov/MeasurementServices/ReferenceMaterials/PUBLICATIONS.cfm.

[4] ISO; *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st ed.; International Organization for Standardization: Geneva, Switzerland (1993); 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://physics.nist.gov/Pubs/.

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; e-mail srminfo@nist.gov; or via the Internet at http://www.nist.gov/srm.

Certificate Revision History: 15 January 2009 (Information value was added for cyanide): 11 October 2007 (Original certificate date).

[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.