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: A certified value for mercury, 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/JCGM Guide [4]. The coverage factor, $k = 1.97$, corresponds to a *t*-factor for approximately 360 degrees of freedom. The measurand is the total mass fraction of mercury reported on a dry mass basis. Metrological traceability is to the SI unit of mass, expressed as milligrams per kilogram.

Table 1. Certified Mass Fraction Value (Dry-Mass Basis) of Mercury

Mercury (Hg)	688 mg/kg	±	28 mg/kg

Reference Value: A reference value for gold, 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 uncertainty 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/JCGM Guide [4]. The coverage factor, $k = 1.98$, corresponds to a *t* factor for approximately 110 degrees of freedom. The measurand is the mass fraction for gold reported on a dry mass basis as determined by ICP-MS. Metrological traceability is to the SI unit of mass, expressed as milligrams per kilogram.

Table 2. Reference Mass Fraction Value (Dry-Mass Basis) for Gold

Gold (Au)	28.0 mg/kg	±	1.5 mg/kg

Information Value: An information value for cyanide is provided in Table 3. An information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to adequately assess the uncertainty associated with the value, or it is a value derived from a limited number of analyses. Information values cannot be used to establish metrological traceability. The cyanide determination was performed by a commercial laboratory using EPA method SW-846 9012a and the mass fraction is reported on a dry mass basis. The value is given only to provide additional information on the matrix.

Table 3. Information Value (Dry-Mass Basis) for Cyanide

Cyanide (CN^-)	96 mg/kg

Expiration of Certification: The certification of **SRM 2451** is valid, within the measurement uncertainty specified, until **31 December 2027**, provided the SRM is handled and stored 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.

Carlos A. Gonzalez, Chief
Chemical Sciences Division

Gaithersburg, MD 20899
Certificate Issue Date: 06 September 2019
Certificate Revision History on Last Page

Steven J. Choquette, Director
Office of Reference Materials

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive changes occur that affect the certification, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of the technical measurements for certification of SRM 2451 was performed by W.R. Kelly formerly of the NIST Chemical Sciences Division.

Analytical measurements at NIST were performed by S.E. Long, A.F. Marlow, and J.R. Sieber of the NIST Chemical Sciences Division, J.L. Mann of the NIST Radiation Physics Division, and W.R. Kelly.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Office of Reference Materials.

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 Office of Reference Materials. After bottling, homogeneity testing was performed by A.F. Marlow 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.

[1] Certain commercial instruments, materials, or processes 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 instruments, materials, or processes identified are necessarily the best available for the purpose.

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; U.S. Government Printing Office: Washington, DC (2000); available at https://www.nist.gov/sites/default/files/documents/srm/SP260-136.PDF (accessed Sep 2019).

[4] JCGM 100:2008; *Guide to the Expression of Uncertainty in Measurement*; (GUM 1995 with Minor Corrections), Joint Committee for Guides in Metrology (JCGM) (2008); available at https://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Sep 2019); 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 https://www.nist.gov/pml/nist-technical-note-1297 (accessed Sep 2019).

Certificate Revision History: 06 September 2019 (Change of expiration date; editorial changes); 18 July 2014 (Extension of certification period; editorial changes); 15 January 2009 (Information value was added for cyanide); 11 October 2007 (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 https://www.nist.gov/srm.