National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material® 2448

Mercury in Brominated Activated Carbon

This Standard Reference Material (SRM) is intended for use in the evaluation of chemical methods of analysis for mercury in halogenated activated carbon sorbents. A unit of SRM 2448 consists of 25 g of brominated activated carbon ground to pass a 250 µm (60 mesh) sieve, homogenized, and packaged in an amber glass bottle.

Certified Value: A certified value for mercury, expressed as a mass fraction [1] on a dry-mass basis, is provided in Table 1. The value is based on analyses by a primary method using cold-vapor isotope dilution inductively coupled plasma mass spectrometry (CV-ID-ICP-MS) [2,3]. 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]. The uncertainty of the certified value is given as an expanded uncertainty $U = ku_c$ about the mean calculated to cover the measurand with approximately 95 % confidence. The quantity u_c is intended to represent, at the level of one standard deviation, the combined standard uncertainty associated with random measurement variability and additional Type B uncertainty components, calculated in a manner consistent with the ISO/JCGM Guide [5], using a coverage factor, $k = 2$. The measurand is the total mass fraction of mercury. The certified value is metrologically traceable to the SI unit of mass, expressed in milligrams per kilogram on a dry-mass basis [1]

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

Mercury (Hg)	0.838 mg/kg ± 0.022 mg/kg

Information Value: An information value for the mass fraction of bromine is provided in Table 2. 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, or only a limited number of analyses were performed [3]. Information values cannot be used to access metrological traceability.

Table 2. Information Mass Fraction Value (As-Received Basis)

Bromine (Br)	3 %

Expiration of Certification: The certification of **SRM 2448** is valid, within the measurement uncertainty specified, until **01 October 2019**, provided the SRM is handled and stored in accordance with instructions given in this certificate (see "Instructions for Handling, Storage, and 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 before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet or register online) will facilitate notification.

Coordination of the technical measurements leading to certification of this SRM was provided by S.E. Long of the NIST Chemical Sciences Division. Analytical measurements leading to certification were made by B.L. Catron, S.E. Long, and J.M. Ness of the NIST Chemical Sciences Division.

Statistical analyses were performed by J.H. Yen of the NIST Statistical Engineering Division.

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Initial processing of the material was performed by C. Campbell and L. Bray at SaskPower, Regina, Saskatchewan, Canada. Particle size reduction, blending and bottling were performed by S.A. Wilson of the United States Geological Survey, Denver, CO, and by M.P. Cronise and C.N. Fales of the NIST Office of Reference Materials.

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

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE

Handling: In addition to mercury, the material may contain other constituents of unknown toxicity. Therefore caution and care should be exercised during its handling and use.

Storage: The SRM should be stored in its original amber bottle, tightly sealed and away from sunlight and intense sources of radiation, under normal laboratory conditions.

Use: Prior to removal of test portions for analysis, the contents should be mixed thoroughly by carefully inverting and rotating the tightly sealed bottle. A minimum test portion mass of 50 mg for mercury should be used for analytical determinations.

Drying Instructions: To relate their measurements directly to the certified and information values that are expressed on a dry-mass basis, users should determine a drying correction at the time of the analysis. The correction is determined by oven-drying a separate 1 g sample at 107 °C ± 3 °C to a constant mass or an equivalent technique. The mass loss determined at NIST using this method was approximately 16.0 %. The mass loss determined by the user may be different, depending on ambient conditions when the bottle is sampled.

SOURCE, PREPARATION, HOMOGENEITY AND ANALYSIS[(1)]

Source and Preparation of Material: The source material for SRM 2448 was a coconut based brominated activated carbon with a nominal particle size range of 20 mesh to 50 mesh. The bulk material was transferred to polyethylene bags and subsequently exposed to flue gases at a coal-fired electric utility plant in Saskatchewan, Canada using a custom built stainless steel exposure vessel connected into a slipstream channel. The exposed material was then ground at the United States Geological Survey using a 40-gallon, corundum-lined, ball mill using one inch corundum grinding media. Approximately 15 kg of material was processed in each of three grinding intervals lasting approximately 100 minutes. The ground material was transferred to NIST and then sieved on a Sweco automated shaker sieve fitted with a 60 mesh screen. The coarse material was discarded and the remaining material blended in a ceramic-lined cone blender for 30 minutes prior to bottling.

Homogeneity Testing: Ten bottles of SRM 2448 were selected for homogeneity assessment of mercury. Duplicate test portions from each bottle were analyzed using a direct combustion atomic absorption spectrometer. Statistical hypothesis tests for differences in the bottle means failed to reject the null hypothesis at the 0.05 significance level for two sample test portion sizes of 50 mg and 150 mg, which is consistent with material homogeneity.

Particle Size Measurements: Particle size measurements for SRM 2448 were made using a Malvern Mastersizer 3000 laser-based light scattering system. Approximately 0.5 g of SRM 2448 (refractive index: 2.42, absorption index: 1.0) material was measured using water as the dispersant, (refractive index: 1.33). Sample was introduced into the measurement cell until an obscuration rate between 5 % and 9 % of the laser beam was achieved. Ten measurements were made in triplicate from each of three bottles. The calculated 10th percentile ($d_{0.1}$), 50th percentile ($d_{0.5}$) and 90th percentile ($d_{0.9}$) particle sizes (percent volume of particles smaller than the value) are $d_{0.1}$ = 5.34 µm, $d_{0.5}$ = 53.5 µm, and $d_{0.9}$ = 140 µm. The fraction of material smaller than 10 µm in diameter is approximately 17 %. The particle size distribution is shown in Figure 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.

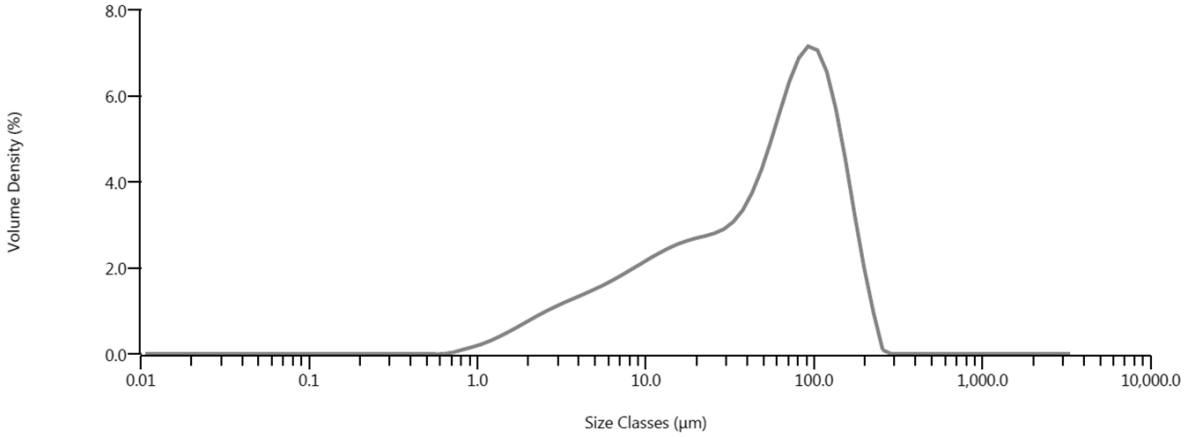

Figure 1. Particle size distribution in SRM 2448

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://www.nist.gov/pml/pubs/index.cfm/ (accessed Jan 2015).

[2] 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).

[3] 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).

[4] 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 Special Publication 260-136, U.S. Government Printing Office: Washington, DC (2000); available at http://www.nist.gov/srm/publications.cfm (accessed Jan 2015).

[5] 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 (2008); available at http://www.bipm.org/utils/common/documents/jcgm/JCGM_100_2008_E.pdf (accessed Jan 2015). 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/tn1297/index.cfm (accessed Jan 2015).

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.