NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY
U.S. DEPARTMENT OF COMMERCE

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Standard Reference Material® 2448
Mercury in Brominated Activated Carbon
CERTIFICATE OF ANALYSIS

Purpose: The certified value delivered by this Standard Reference Material (SRM) is intended for use in the evaluation of chemical methods of analysis for mercury in halogenated activated carbon sorbents.

Description: 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 wet-mass (as-received) basis, is provided below. 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 about the mean to cover the measurand with approximately 95 % confidence. The expanded uncertainty is calculated as $U = ku_c$ where u_c is the combined standard uncertainty consistent with the ISO/JCGM Guide [5], and k is a coverage factor, ($k = 2$) corresponding to approximately 95 % confidence. The certified value is metrologically traceable to the International System of Units (SI) unit of mass fraction expressed in milligrams per kilogram (mg/kg) on a wet-mass basis [1].

Certified Mass Fraction Value (Wet-Mass Basis)

Mercury (Hg)	0.723 mg/kg ± 0.049 mg/kg

Additional Information: Values of potential interest to users and additional information are provided in Appendices A and B.

Period of Validity: The certified value delivered by **SRM 2448** is valid within the measurement uncertainty specified until **31 July 2034**. The certified value is nullified if the material is stored or used improperly, damaged, contaminated, or otherwise modified.

Maintenance of Certified Values: NIST will monitor this SRM over the period of its validity. If substantive technical changes occur that affect the certification, NIST will issue an amended certificate through the NIST SRM website (https://www.nist.gov/srm) and notify registered users. SRM users can register online from a link available on the NIST SRM website or fill out the user registration form that is supplied with the SRM. Registration will facilitate notification. Before making use of any of the values delivered by this material, users should verify they have the most recent version of this documentation, available through the NIST SRM website (https://www.nist.gov/srm).

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Safety: 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. The material is moderately hygroscopic; therefore, precautions should be taken to minimize exposure to humid environments. Do not leave the bottle open for longer than is necessary to sub-sample the material. After use replace the cap tightly prior to storage. A minimum test portion mass of 50 mg should be used for analytical determinations of mercury mass fraction.

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 https://www.nist.gov/pml/special-publication-811 (accessed Oct 2024).

[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] Beauchamp, C.R.; Camara, J.E.; Carney, J.; Choquette, S.J.; Cole, K.D.; DeRose, P.C.; Duewer, D.L.; Epstein, M.S.; Kline, M.C.; Lippa, K.A.; Lucon, E.; Molloy, J.; Nelson, M.A.; Phinney, K.W.; Polakoski, M.; Possolo, A.; Sander, L.C.; Schiel, J.E.; Sharpless, K.E.; Toman, B.; Winchester, M.R.; Windover, D.; *Metrological Tools for the Reference Materials and Reference Instruments of the NIST Material Measurement Laboratory*; NIST Special Publication (NIST SP) 260-136, 2021 edition; National Institute of Standards and Technology, Gaithersburg, MD (2021); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-136-2021.pdf (accessed Oct 2024).

[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 https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Oct 2024). 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 Oct 2024).

Certificate Revision History: 09 October 2024 (Change of period of validity; updated format; editorial changes); 23 January 2020 (Changed the certified mercury value to a wet mass value; editorial changes); 15 July 2019 (Change of expiration date; editorial changes); 07 January 2015 (Original certificate date).

Certain commercial equipment, instruments, or materials may be identified in this Certificate of Analysis 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.

Users of this SRM should ensure that the Certificate of Analysis in their possession is current. This can be accomplished by contacting the Office of Reference Materials 100 Bureau Drive, Stop 2300, Gaithersburg, MD 20899-2300; telephone (301) 975-2200; e-mail srminfo@nist.gov; or the Internet at https://www.nist.gov/srm.

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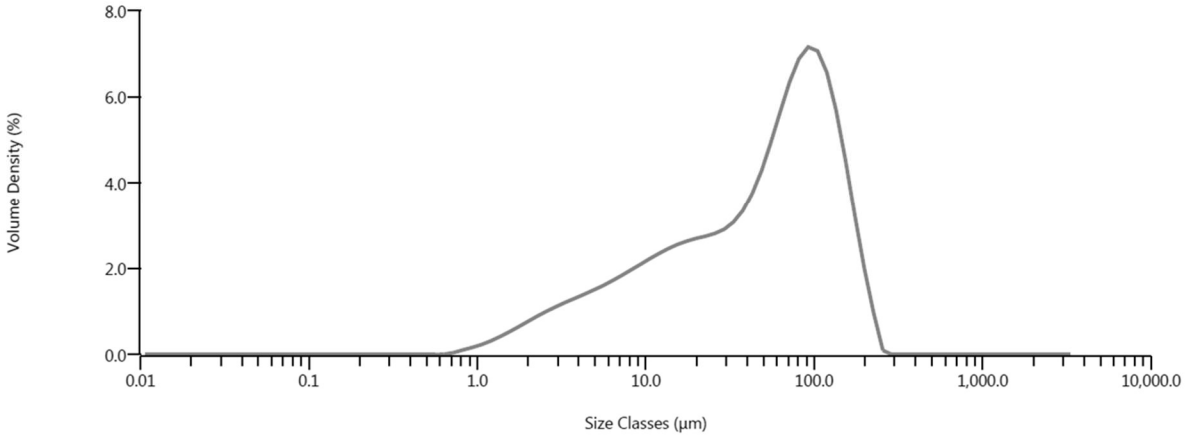
APPENDIX A

Values of Potential Interest to Users: Values of potential interest to users for moisture, particle size, and mass fraction of bromine are provided below. A value of potential interest to users 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]. Values of potential interest cannot be used to establish metrological traceability.

Moisture: The moisture content was determined at NIST by oven drying a 1 g sub-sample at 107 °C to constant mass. The moisture content of SRM 2448 is approximately 16 %.

Particle Size: 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 the figure below.



Particle size distribution in SRM 2448

The value of potential interest to users mass fraction value given below is provided without uncertainty estimates and is given as additional information on the matrix.

Value of Potential Interest to Users Mass Fraction (Wet-Mass Basis)

Bromine (Br)	3 %

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APPENDIX B

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.

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