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

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Standard Reference Material® 1641f
Mercury in Water
CERTIFICATE OF ANALYSIS

Purpose: The certified value delivered by this Standard Reference Material (SRM) is intended for the calibration of instruments and techniques used for the determination of mercury in natural waters. It is designed for the preparation of calibration solutions and for use as a "spike" sample in a "method-of-additions" analytical procedure.

Description: A unit of SRM 1641f consists of 10 sealed borosilicate glass ampoules. Each ampoule contains approximately 10 mL of solution consisting of a trace amount of mercury in approximately 3 % mass fraction nitric acid and 2 % mass fraction hydrochloric acid.

Certified Values: The certified value for mercury in SRM 1641f is shown below. 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 accounted for by NIST [1]. The certified value is metrologically traceable to the International System of Units (SI) derived unit for mass fraction, expressed as milligrams per kilogram.

Certified Mercury Mass Fraction: 0.1013 mg/kg ± 0.0024 mg/kg

The certified value was calculated as the weighted mean of the mass fraction values obtained through (1) analysis by isotope dilution cold-vapor inductively coupled plasma mass spectrometry (ID-CV-ICP-MS) and (2) analysis by direct combustion atomic absorption spectrometry (DC-AAS) [2].

The uncertainty in the certified value is given as an expanded uncertainty $U = ku_c$, where u_c is the combined standard uncertainty calculated according to the ISO/JCGM Guide [3] and k is a coverage factor ($k = 2$) used to obtain an approximate confidence level of 95 %. The value of u_c is intended to represent, at the level of one standard deviation, the combined effect of uncertainty components associated with ID-CV-ICP-MS and DC-AAS measurement [2].

Period of Validity: The certified values delivered by **SRM 1641f** are valid within the measurement uncertainty specified until **28 February 2030**. The certified values are 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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Metrological Traceability: Metrological traceability of measurement results to a given reference must be established through an unbroken chain of calibrations and/or comparisons, each having stated uncertainties [4], using measurement standards that are appropriate for the physical or chemical property being measured. Comparisons may include validation measurements using various spectroscopic or classical methods of analysis. Gravimetric or volumetric dilution is also a method of comparison, where the mass or volume of a solution before and after dilution is measured.

For this SRM, the measurand is the total concentration of mercury expressed as mass fraction, and the certified value is metrologically traceable to the SI unit of mass. This SRM can be used to establish traceability of the results of mercury measurements to NIST measurement results and standards. One approach is to calibrate analytical instruments or procedures for the determination of mercury using standards whose values are traceable to the certified value of mercury in this SRM. When the traceable values of such standards are assigned using this SRM for calibration, the uncertainties assigned to those values must include the uncertainty of the certified value of this SRM, appropriately combined with the uncertainties of all calibration measurements.

Safety: This SRM is an acidic solution contained in tip-sealed borosilicate glass ampoules with pre-scored stems. Therefore, all appropriate safety precautions, including use of gloves during handling, should be taken. Consult the Safety Data Sheet (SDS), enclosed with the SRM shipment, for chemical hazard information.

Storage: Unopened ampoules should be stored under normal laboratory conditions in an upright position inside the original container supplied by NIST.

Use: When an ampoule is to be opened, the area of the stem where the pre-scored band is located (≈ 5 mm below the encircling metallic band) should be carefully wiped with a clean, damp cloth and the body of the ampoule wrapped in absorbent material. Holding the ampoule steady and with thumb and forefinger grasping the stem at the metallic band, **minimal** thumb pressure should be applied to the stem to snap it. Correctly done, the stem should break easily where pre-scored. Use of a metal file to break the stem is **NOT** recommended.

Traces of mercury vapor are present in most laboratory environments. Therefore, contamination of reagents, equipment, and common laboratory materials may cause a severe blank or background problems. Apparatus for analyses at and below the milligram-per-kilogram level must be scrupulously cleaned immediately before use, and only the purest reagents with respect to mercury should be used. Once ampoules are opened, the entire contents should be transferred immediately to another container and dilutions should be prepared and used without delay since stability of the dilutions cannot be guaranteed. If desired, this SRM can be used to prepare more dilute working standard solutions. Blank determinations should be made of the diluent reagents. The user should establish internal laboratory procedures that specify a maximum shelf-life for a working standard solution. Two procedures for the preparation of working standard solutions follow.

Preparation of Standard Solutions by Mass: Diluted working standard solutions can be prepared by transferring an aliquot of the SRM to an empty, dry, pre-weighed polyethylene bottle, and then reweighing the bottle. An appropriate dilute acid must be added by mass to bring the solution to the approximate desired dilution. The dilution need not be exact since the mass of the empty bottle, mass of the bottle plus SRM aliquot, and the final diluted mass of the solution will permit calculation of the exact mass fraction (mass of mercury per mass of solution) of the working standard solution. Dilutions prepared gravimetrically as described will need no correction for temperature and no further correction for true mass fraction in vacuum.

Preparation of Standard Solutions by Volume: Volumetric dilutions are NOT recommended due to uncertainties in volume calibrations and variations in density. If dilutions must be made volumetrically, then they may be made by the addition of accurately measured aliquots, withdrawn from the just opened ampoule, to known volumes of an appropriate dilute acid using conventional techniques. The volumetric apparatus used should be scrupulously cleaned. The reliability of the dilution process will depend on the care exercised and on the reliability of the calibration of the volumetric apparatus used.

Possible Presence of Other Elements: Studies conducted by NIST have shown that components of borosilicate glass ampoules may leach into solution. In undiluted solutions, Na and Si mass fractions as large as 20 mg/kg, B and La mass fractions in the range 1 mg/kg to 5 mg/kg, and Al, As, Ce, Mg, Mn, Rb, and Zn mass fractions in the range 0.05 mg/kg to 1 mg/kg have been found. Possible effects should be considered when this SRM is used.

NOTICE TO USERS

NIST strives to maintain the SRM inventory supply, but NIST cannot guarantee the continued or continuous supply of any specific SRM. Accordingly, NIST encourages the use of SRMs as primary benchmarks for the quality and accuracy of the user's in-house (working) standards. As such, SRM should be used to validate the more routinely used standards in a laboratory. Comparisons between the SRM and in-house reference materials or working measurement standards should take place at intervals appropriate to the conservation of the SRM and the stability of relevant in-house materials. For further guidance on how this approach can be implemented, contact NIST by email at srms@nist.gov.

Full details on the production and evaluation of SRM 1641f are provided free of charge in reference 2**.**

REFERENCES

[1] 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 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 Mar 2025).

[2] Bryan Sallee CE; Butler TA; Klingsick JR; Molloy JL; Yen JH; *Certification of Standard Reference Material® 1641f Mercury in Water.* NIST Special Publication (SP) 260-250. National Institute of Standards and Technology, Gaithersburg, MD, (2024); available at https://nvlpubs.nist.gov/nistpubs/SpecialPublications/NIST.SP.260-250.pdf (accessed Mar 2025).

[3] JCGM 101:2008; *Evaluation of Measurement Data — Supplement 1 to the "Guide to the Expression of Uncertainty in Measurement" — Propagation of Distributions using a Monte Carlo Method*; Joint Committee for Guides in Metrology (JCGM) (2008) available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Mar 2025).

[4] JCGM 200:2012; *International Vocabulary of Metrology - Basic and General Concepts and Associated Terms*, 3rd ed.; Joint Committee for Guides in Metrology (JCGM) (2012); available at https://www.bipm.org/en/committees/jc/jcgm/publications (accessed Mar 2025).

If you use this SRM in published work, please reference:

Bryan Sallee CE, Butler TA, Klingsick JR, Molloy JL, Yen JH (2024) Certification of Standard Reference Material® 1641f Mercury in Water. (National Institute of Standards and Technology, Gaithersburg, MD), NIST Special Publication (SP) 260-250. https://doi.org/10.6028/NIST.SP.260-250

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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