

U. S. Department of Commerce
Frederick B. Dent
Secretary
National Bureau of Standards
Richard W. Roberts, Director

National Bureau of Standards Certificate of Analysis Standard Reference Material 1641 Mercury in Water - Concentrate

This Standard Reference Material is intended for use in the primary calibration of instruments and techniques used for the analysis of mercury in natural waters. It is designed for the preparation of calibration solutions or for use as a "spike" sample in a "method-of-additions" type analytical procedure. Normally a dilution of one part standard to 1000 parts of natural water will be used.

Mercury concentration $1.49 \pm 0.05 \mu\text{g/ml}$

The estimate of accuracy shown is believed to express the overall uncertainty of the certified value. It includes the standard error of the weighted average of 35 determinations by three different analytical techniques, allowances for within-method and between-method variability, and allowances for possible systematic errors.

Stability: Trace mercury solutions have been a constant problem when long-term storage is required. At or below the $\mu\text{g/ml}$ level, mineral acid stabilization is not sufficient. A new stabilizing technique has been applied to this Standard Reference Material which allows for prolonged storage. Gold, as the tetrachloride, has been added in a concentration 10 times that of mercury. The gold ion, in conjunction with the normal mineral acid, has proven an effective stabilizer. Stabilization studies have been carried out for nine months and are continuing. It is recommended that this Standard Reference Material not be used after ONE YEAR FROM DATE OF PURCHASE.

Precautions: Traces of mercury vapor are present in most laboratory situations. Therefore, contamination of reagents, equipment, and common laboratory materials may cause a severe problem. Apparatus for analysis at this level must be scrupulously cleaned immediately before use, and only the purest-grade reagents employed. When possible, a Class-100 clean room should be used for all samplings and manipulations.

Washington, D. C. 20234
March 5, 1975

J. Paul Cali, Chief
Office of Standard Reference Materials

(over)

This Standard Reference Material was prepared by J. R. Moody. Atomic absorption analyses were performed by T. C. Rains, isotope-dilution mass spectrometry by J. R. Moody and P. J. Paulsen, and neutron activation analyses by H. L. Rook.

The overall direction and coordination of the technical measurements leading to the certification were performed under the chairmanship of H. L. Rook.

The technical and support aspects involved in the preparation, certification, and issuance of this Standard Reference Material were coordinated through the Office of Standard Reference Materials by T. W. Mears.

Analytical: Three independent techniques were used in the certification of this Standard Reference Material: atomic absorption spectroscopy, isotope dilution-spark source mass spectrometry, and neutron activation analysis. Sample sizes for analysis varied from 1 to 20 ml, depending on the technique used. The mean results obtained by the individual techniques were in agreement within a coefficient of variation of two percent.

Use: The SRM as issued consists of 6 ampoules, each containing approximately 20 ml of solution. Dilutions may be made by addition of accurately measured aliquots, withdrawn from an ampoule, to known volumes of pure or to natural water (spiking mode) using conventional techniques. Blank determinations should be made of the water used.

The reliability of the dilution process will depend on the care exercised and the reliability of the calibration of the volumetric apparatus, which should be within one percent to be consistent with the accuracy of this reference material. The volumetric ware should be scrupulously cleaned; and the solutions prepared should be used without undue delay, as their stability cannot be certified.

The long term retention of unused portions of this Standard Reference Material in opened ampoules is not recommended.