

National Bureau of Standards

Certificate of Analysis

Standard Reference Material 1630

Trace Mercury in Coal

This Standard Reference Material is intended as an analytical standard for the determination of trace mercury in coal. The material is a commercially available coal that was crushed to a size of 210 to 500 micrometers with a roll crusher. From a total of 500 packaged bottles, 30 were randomly selected for analysis. Duplicate determinations were made on 0.5 g portions of 25 of these bottles, and single determinations were made on the other five. The mercury content of this material was obtained by destructive neutron activation analysis.

The recommended value is the average of these 55 determinations on 30 bottles, which was found to be:

Mercury content = 0.13 parts per million (by weight)

Based upon work done thus far, it is expected that the final certified value, when obtained, will not change by more than plus or minus one (1) in the last significant figure of the provisionally recommended value given above.

A study of homogeneity showed no variability among bottles that could not be accounted for by analytical error. The standard deviation between duplicate samples from the same bottle was 0.0064 ppm corresponding to a coefficient of variation of 5.0%.

The mercury content was also determined by flameless atomic absorption spectrometry, yielding an average value of 0.14 parts per million.

Selenium was also determined using destructive neutron activation analysis. The value obtained, which is not certified but included for information only, was found to be 2.1 parts per million.

The homogeneity testing and analyses for certification were performed in the NBS Analytical Chemistry Division by T. E. Gills and H. Rook under the direction of P. D. LaFleur.

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 C. L. Stanley.

Washington, D. C. 20234
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J. Paul Cali, Chief
Office of Standard Reference Materials

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ANALYTICAL PROCEDURE

The bottles containing the samples were allowed to remain open at room temperature (about 25 °C) for twenty-four hours.

The coal samples, along with standards for mercury solutions and NBS Standard Reference Material 1571 (Orchard Leaves) used as controls, were encapsulated in cleaned quartz vials. The geometry of both the samples and the standards were optimized so that flux monitors were not needed. The samples were irradiated for four hours at a thermal flux of $5 \times 10^{13} \text{ N}\cdot\text{cm}^{-2} \text{ sec}^{-1}$. The samples were allowed to decay for three days to minimize the personnel dose rate. The samples were postweighed into porcelain boats and burned in a combustion tube. The volatile mercury compounds and other volatile products liberated during burning were trapped in a liquid nitrogen cold trap. The cold trap was allowed to warm to room temperature. The mercury compounds were then transferred to polyethylene bottles by washing the cold trap with concentrated nitric acid and water. For this analysis, ^{197}Hg produced by $^{196}\text{Hg}(\text{n},\gamma)^{197}\text{Hg}$ was used as the measuring activity.

Bromine-82, an interfering isotope, was separated from the sample by using the classical silver bromide precipitation.

The samples were counted on a 22 cm³ Ge(Li) detector equipped with a 2048-multichannel analyzer. The accumulated data was processed by computer for peak identification and integration. The concentrations were determined by using a Standard Comparator Method.

NOTE TO USER

It is suggested that persons using SRM 1630 to check their analytical technique adopt the following criteria: If the average, \bar{X} , of N replicate measurements on this SRM is found to lie in the interval—

$$0.127 - \frac{0.013}{\sqrt{N}} < \bar{X} < 0.127 + \frac{0.013}{\sqrt{N}}$$

then the analytical technique used gives a result compatible with that found at NBS. However, if the value \bar{X} lies outside this interval, then the technique should be examined for possible bias or miscalibration.

[NOTE: The above expression is not rigorously correct. It does not include a possible component for between laboratory variability nor sources of systematic error. An NBS 260 Special Publication to discuss these and related matters is in preparation.]